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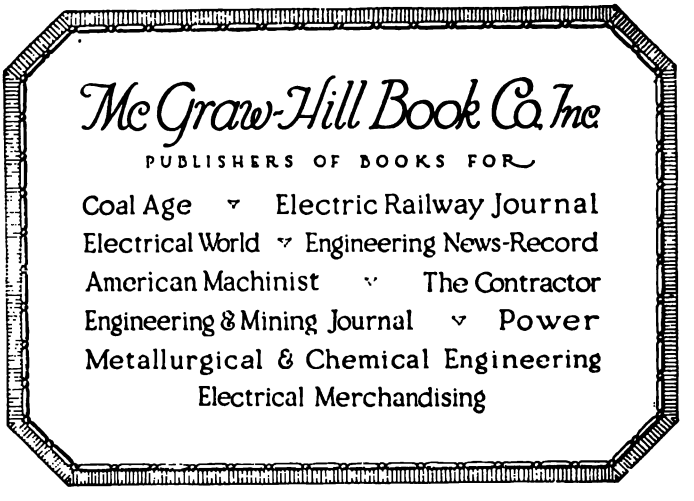
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# THE AMERICAN PETROLEUM INDUSTRY

## VOLUME II


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# THE AMERICAN
# PETROLEUM INDUSTRY

## VOLUME II

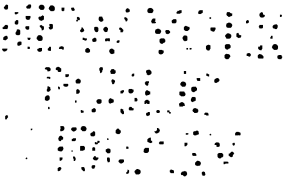
BY

RAYMOND FOSS BACON, PH. D.,
DIRECTOR OF THE MELLON INSTITUTE OF INDUSTRIAL RESEARCH OF THE UNIVERSITY OF PITTSBURGH

AND

WILLIAM ALLEN HAMOR, M. A.,
ASSISTANT DIRECTOR OF THE MELLON INSTITUTE OF INDUSTRIAL RESEARCH

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# THE AMERICAN PETROLEUM INDUSTRY

## VOLUME II

### CHAPTER XII

### REFINERY TECHNOLOGY

From the viewpoint of the petroleum refiner, crude oil is of two general classes, or types, namely: those petroleums which carry practically no asphalt and which yield solid hydrocarbons of the paraffin series ($C_nH_{2n+2}$), and termed "paraffin-base;"[1] and those which yield practically no solid paraffins, but are rich in asphalt, the "asphalt-base" petroleums.[2] Strictly speaking, however, there is still another class, the "paraffin-asphalt," "semi-asphalt," or mixed-base petroleums, typified by certain of the crude oils of Illinois, Kansas, Oklahoma, and northern Texas, which contain both paraffin and asphalt,[3] and are hence a combination of the two other classes.

As obtained by drilling through the overlying strata to the oil sands beneath,[4] petroleum is invariably accompanied by water and "bottom settlings" ("B.S."). The water is removed by settling or other suitable procedure.[5] Settling also serves for the elimination of the mineral matter and inert organic substances composing some "B.S.;"[6] but if the latter consists of an emulsion of amorphous paraffin wax and water, as is sometimes the case

[1] The petroleum of the Appalachian field (Pennsylvania, New York, West Virginia, southeastern Ohio, and Kentucky) is "paraffin-base;" it contains practically no asphaltic matter and very little sulphur (0 06 to 0.08 per cent.). The crude oils from Lima, Ohio, and Canada are also "paraffin-base," but are high in sulphur (see p. 609). Paraffin-base petroleums predominate in northern Louisiana.

PECKHAM (*Pet. Ind. Techn. Rev.*, **3**, *Suppl.*, 39) classifies petroleums into four types: "paraffin," "Russian," "sulphur," and "nitrogen."

On the classification of crude oils, see also GUISELIN, *Mat. grasses*, **3**, 1830; and CHERCHEFFSKY, *ibid.*, **5**, 2640.

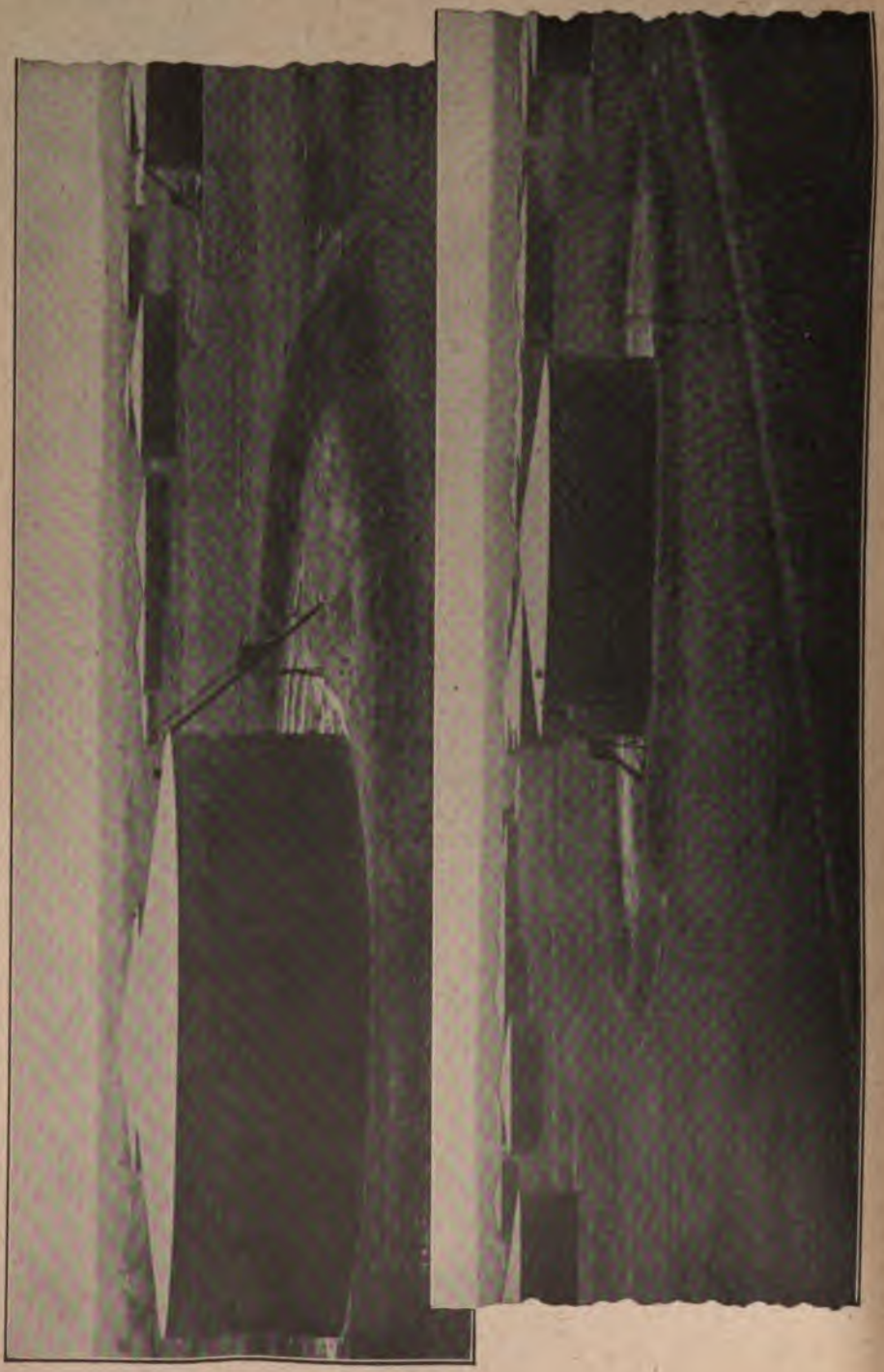
[2] The California crude oils and certain of the Texas crude petroleums are of this class. See pp. 131 and 167.

[3] On the composition of these petroleums, see pp. 132, 139, 157 and 167. The crude petroleum of Mexico belongs to this class. On the Mexican oil fields, see HUNTLEY, *Bull. Am. Inst. Min. Eng.*, **1915**, No. **105**, 2067.

[4] See p. 272.

[5] On the dehydration of petroleum, see p. 528.

[6] See p. 855.



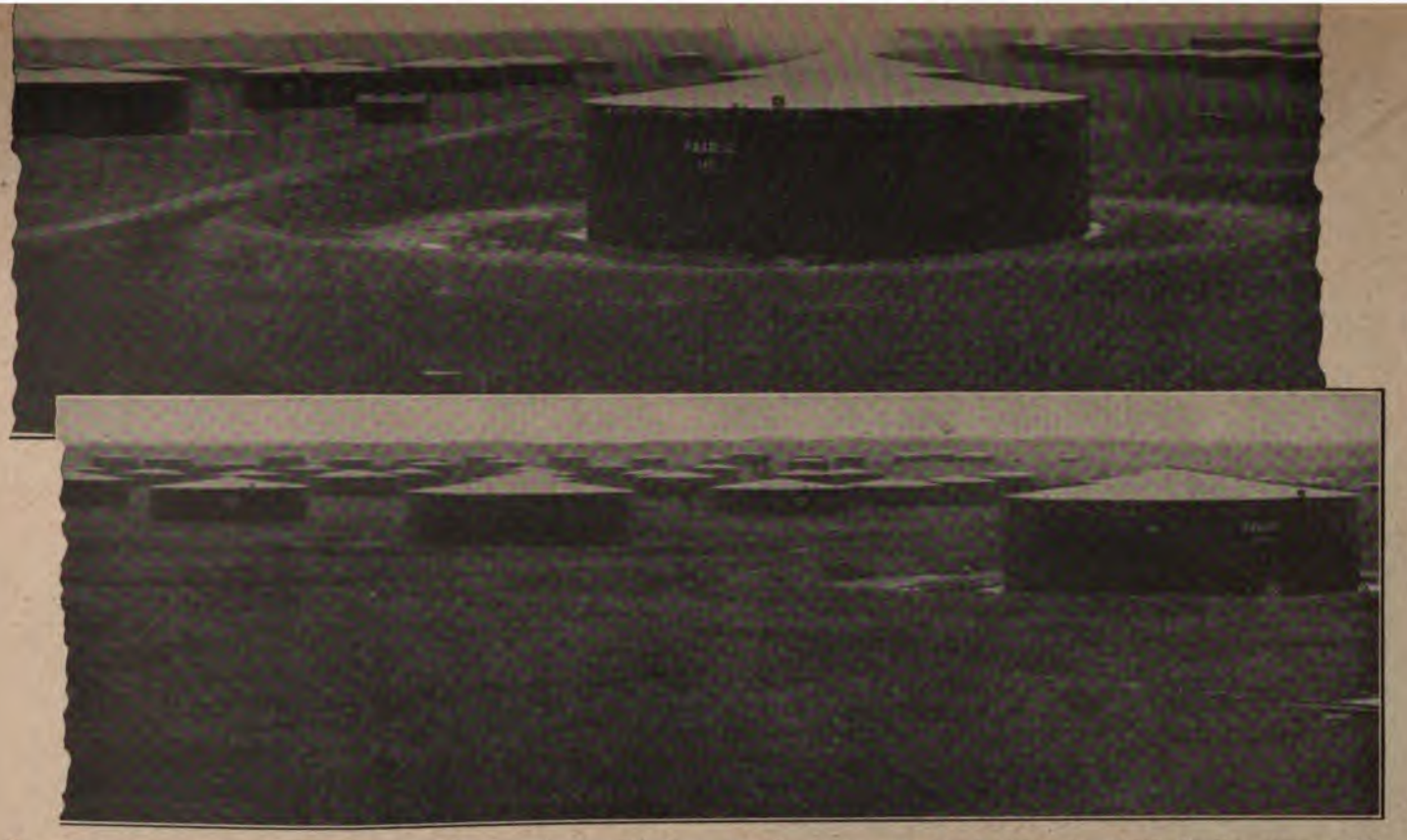


FIG. 156.—A tank farm in the Cushing, Oklahoma, field. This farm contains between 400 and 500 tanks, 115 ft. in diameter and 30 ft. in height.

in the Appalachian field, it is usually refined with the petroleum after the removal of the free water.[1]

Petroleum is transported from the oil fields to the storage tanks of the refineries, either through pipe lines of from generally 4 to 8 in. in diameter[2] or by tank cars. At the present time, petroleum from Oklahoma is being transported by pipe lines, via Kansas City and Chicago, to the Atlantic seaboard, a distance of about 1,600 miles.



FIG. 157.—A tank farm in the Caddo District, Louisiana. These tanks are 95 ft. in diameter and 30 ft. in height; each has a capacity of 37,500 barrels.

The more viscous varieties of petroleum[3] are sometimes used in the crude state for fuel,[4] for surfacing roads,[5] or for the prevention of dust on roads. However, ordinarily the first stage in the utilization of petroleum for any purpose is to subject it

[1] Cf. the utilization of "B.S." in the manufacture of petrolatum, p. 632.

[2] For valuable papers on the flow of oil through pipes, see MERCZYNG, *Chem.-Ztg. Repert.*, **15** (1891), 223; and ERBICEANU, *Chem. Tech. Ztg.*, **29**, 106. On pipe line construction, see H. W. CROZIER, *J. Elec. Power and Gas*, **35** (1915), 161–78.

[3] Such oils contain, as a rule, considerable asphalt and sufficient water (cf. p. 528) to render their distillation difficult. At present not more than one-fourth of the petroleum produced in the United States finds any utilization in the natural state.

[4] Petroleums used in the crude state as fuel are the more viscous varieties occurring in California and Mexico. About 30 per cent. of the fuel oil burned in Mexico in 1913 was crude, untreated petroleum.

[5] Certain of the oils used on roads are the heavier grades containing asphalt, which, in the case of the California crude oils, sometimes reach 60 per cent. Near Lander, Wyoming, is found a petroleum so viscous that it can be used for roads without refining.

to some form of refining which will reduce it to the most desirable consistency for the purpose intended.

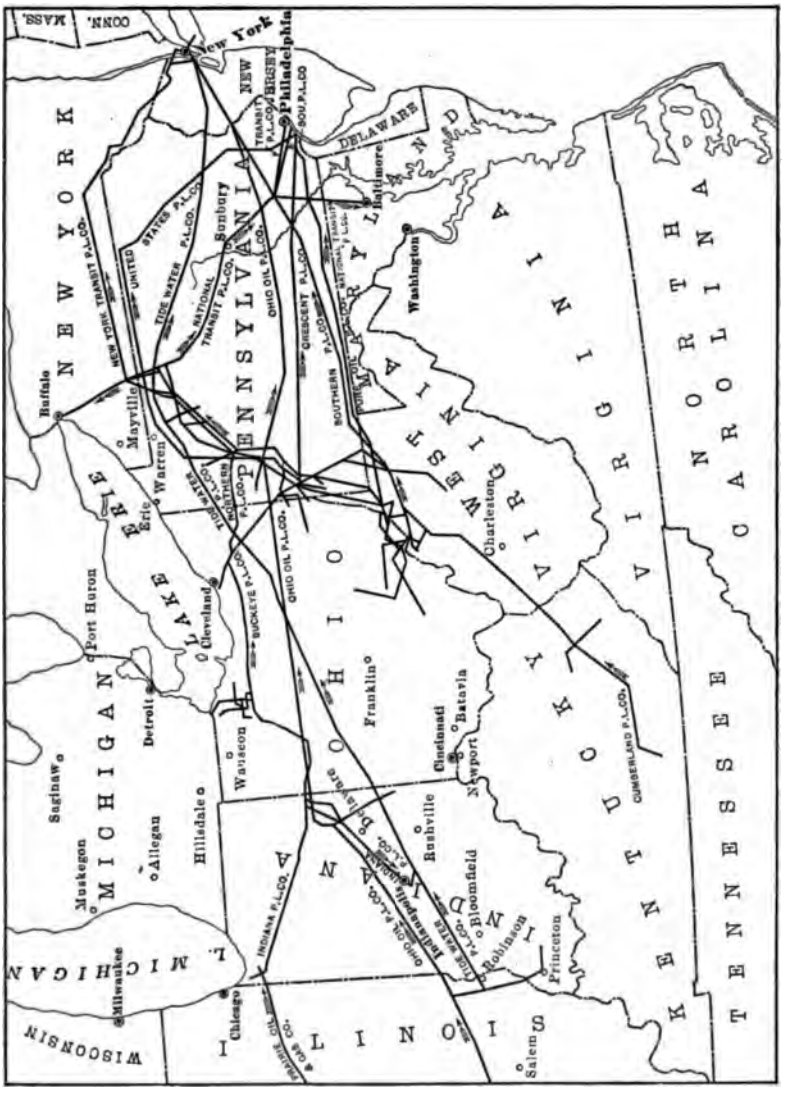



FIG. 158.—Pipelines in the Appalachian Field.

## THE REFINING OF PETROLEUM

Small amounts of the crude oils obtained in Pennsylvania and West Virginia possess lubricating qualities *per se*, that is,

in their natural state, and are employed as lubricants under the name of "natural oils," either without any treatment, or after straining, clarification by settling, or filtration through fullers'




FIG. 159.—A tank car of the type now in use.

earth or bone-black; but, since the production is small, these oils are of mere passing interest. Then, too, at one time, be-

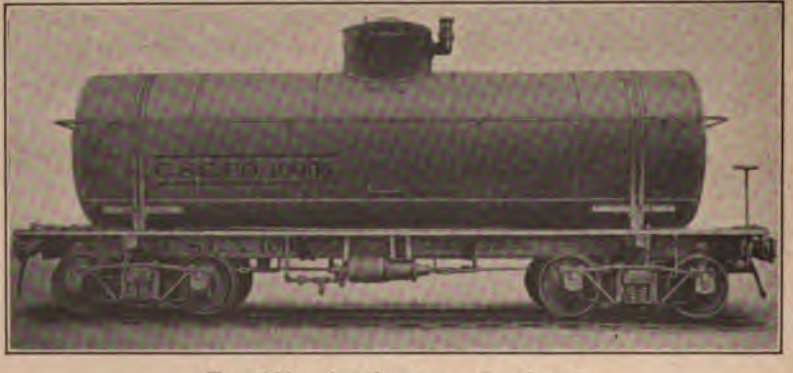


FIG. 160.—Another type of tank car.

fore a demand for light distillates existed, certain petroleums were deprived of a part of their more volatile constituents by

atmospheric evaporation; the production of such "reduced" oils is no longer practised[1] to the knowledge of the authors.

TABLE XLII.—THE QUANTITY OF CRUDE PETROLEUM USED BY AMERICAN REFINERIES AND THE VALUES OF THE VARIOUS PRODUCTS MANUFACTURED

| | 1909 | | 1914 | |
|---|---|---|---|---|
| | Barrels | Value | Barrels | Value |
| No. of establishments | 147 | | 176 | |
| Total production of crude petroleum | 183,170,874 | ..... | 265,762,535 | |
| Crude petroleum used | 120,775,439 | $152,307,040 | 191,262,724 | $249,727,855 |
| PRODUCTS | | | | |
| Naphthas and lighter products: gasoline (from crude petroleum) | 10,806,550 | 39,771,959 | 23,908,242 | 106,140,170 |
| All others | ..... | ..... | 5,292,522 | 15,779,137 |
| Illuminating oils | 33,495,798 | 94,547,010 | 38,705,496 | 96,806,452 |
| Fuel oils | 34,034,577 | 36,462,883 | 74,669,821 | 84,017,916 |
| Lubricating oils | 10,745,885 | 38,884,236 | 10,348,521 | 55,812,120 |
| Residuum or tar, including liquid asphaltic road oils | 1,787,008 | 2,215,623 | 2,696,887 | 4,017,858 |
| Greases | 138,302 | 1,567,647 | 280,128 | 3,536,491 |
| Paraffin wax | 946,830 | 9,388,812 | 1,150,776 | 8,897,106 |
| Asphalt, other than liquid asphalt—tons (2000 lbs.) | 233,328 | 2,724,752 | 465,157 | 4,867,213 |
| All other products | | 11,434,737 | | 16,486,942 |
| Total value of products | ..... | $236,997,659 | ..... | $396,361,405 |

Generally, the first step in the separation of crude oil into its various products[2] is fractional distillation, which may be modified according to the nature of the petroleum and the products desired.[3] The distillates obtained are usually purified by treatment, successively, with sulphuric acid and a solution

[1] See p. 527.

[2] Gasolines, Naphthas, Kerosenes, Gas Oil, Fuel Oil, Spindle Oils, Cylinder Oils, Paraffin Wax, Petrolatum, Dust Laying Oils, Road Binders, and Coke. The various products of petroleum are defined in Chap. XVIII. For an excellent account of the manufacture of petroleum products, see F. C. ROBINSON, *Met. Chem. Eng.*, **11** (1913), 389–94. On European refining methods, see SEELENFRIED, *Western Eng.*, Dec., **1915**; and WOLF, *Chem. App.*, **2** (1915), 259 and 284.

[3] It may be mentioned here that the products of fractional distillation, even on a laboratory scale, are not identical with the hydrocarbons present in the petroleum, since they are in part the result of the action of heat upon these hydrocarbons. On this point, see THORPE and YOUNG, *Proc. Roy. Soc.*, **19** (1871), 370; **20** (1872), 488. On the distillation of petroleum, see GURWITSCH'S "Wissenschaftliche Grundlagen der Erdölbearbeitung," **1913**, part ii.

of sodium hydroxide, or what is commonly called caustic soda, each followed by washing with water.[1]

The apparatus used for the operation of distillation is described in detail in another chapter.[2] Suffice it to say here that the stills[3]



FIG. 161.—The front of the firing-end of a battery of crude stills, 10 ft. in diameter and 40 ft. in length.

[1] For the early history of petroleum refining in the United States, see p. 228. On the chemical treatment of petroleum distillates, see pp. 579 to 609. On agitators, see p. 744. On explosions in agitators, see p. 788.

[2] See p. 644.

[3] The stills employed in petroleum refineries are of the following general types: *Crude Stills*, wherein crude petroleum is run down to tar. It was once the practice to redistil some distillates in stills of this type, but at the present time reduction is generally carried out in *Steam Stills*, the gasolines and illuminating oils being reduced therein to the desired tests. Steam is also used in the fractional distillation of crude petroleum, in which procedure (see *infra*) it is introduced for the production of steam-refined cylinder stocks. The tar resulting from the distillation of petroleum in crude stills is cracked at higher temperatures in *Tar Stills* for the production of paraffin wax and paraffin engine oils. In *Lubricating Stills* the crude petroleum is run, with the assistance of bottom steam, to take off the natural burning oil and wax distillate, and to convert the remainder into lubricating oils. Other types of stills are *Tower Stills*, stills of the ordinary type provided with dephlegmating columns; *Vacuum Stills*, referred to below; and *Continuous Stills*, which are employed when petroleum is distilled continuously. For a general account of stills, see p. 716.

employed are usually horizontal steel shells set in brickwork[1] and connected at their domes with vapor pipes which lead to condensers, consisting of coils of pipe set in tanks through which water is circulated.[2] Pipes termed "running lines" connect the condenser coils with the "tail house," where the "running lines" are intercepted by "look boxes,"[3] arranged so that the distillate flowing from the condenser (the "stream") may be watched and sampled, and connected at their bottoms with manifolds having valves for running the "stream" into the desired receiving tank. The distillation process is generally controlled by the gravity of the "stream," as determined by the stillman in charge. The uncondensed gas issuing from the condensers is separated by means of traps[4] on the "running lines;" this gas is subjected to pressure, in order to separate the light gravity gasoline, and the residual gas is used as a refinery fuel.[5]

It is now the practice at a number of large American refineries to distil petroleum by the *continuous system*,[6] as distinguished from the intermittent method of distillation. The stills are connected and operated in a continuous manner, by simultaneously charging the first still in the battery slowly and pumping from the last, and by firing each successive still stronger than the preceding one; by this procedure, fractions possessing different boiling points are obtained simultaneously from each still in the battery.

With regard to the method of distilling in use, it may first be said that the *"dry" or destructive ("cracking") distillation process* is used when a large yield of gasoline and illuminating oil is desired. It is better adapted to petroleum which is unfit for the manufacture of cylinder stocks. In this process, the crude oil is fire-stilled, and the heavy vapors which condense in the top of the still fall back into the superheated oil and are thereby "cracked," or partially decomposed.[7] In this way,

[1] See p. 722.

[2] See p. 735 for a general description of condensers.

[3] These are described on p. 772.

[4] See p. 775.

[5] Cf. the practice at the Scottish shale-oil works, p. 833.

[6] For a full account of continuous distillation, see p. 533. Cf. the Scottish practice referred to on p. 834. On the heat exchangers used when petroleum is distilled continuously, see p. 772.

[7] On the "cracking" of petroleum distillates, see p. 554. For Russian practice, see KHARITCHKOV, *Chem. Rev. Fett- Harz-Ind.*, **17**, 33.

there are produced oils of lighter gravity than would otherwise be obtained at that point. Until recently, a general practice in the large refineries handling paraffin-base petroleum has been to first run the petroleum to residuum[1] in the crude oil stills, and then to distil the residuum, or tar, in the tar stills for wax distillate[2] and coke. Now, however, the distillation is usually continued to the production of coke in tower stills,[3] without



FIG. 162.—Steam stills (10 ft. by 40 ft.), condensers and receiving house at a California refinery. The distillates go directly from the condensers through the receiving house into receiving tanks of the water-top type (see p. 709).

interruption, thus separating the various products (as naphtha, illuminating oils, and paraffin oils) in one distillation.

In the "*fractional distillation*," or distillation with bottom steam, which is employed when it is desired to prevent the decomposition of the petroleum as much as possible, particularly when lubricating oils, such as cylinder oils, are to be manufactured from paraffin-base petroleum, free "dry" steam[4] is introduced into the body of the oil in the still by a coil or branched

[1] The yield of tar, or residuum, by this process amounts to about 11 per cent. of the paraffin-base petroleum distilled.

[2] Wax distillate ("tar distillate") is a lubricating oil distillate of about 30°Bé.; it contains the paraffin wax.

[3] See p. 728 for an account of stills of the dephlegmation type.

[4] The supply of steam is always carefully regulated. On the behavior of high-boiling mineral oils on heating in air and steam, see SCHREIBER, *Z. angew. Chem.*, **23**, 99.

pipe so perforated that the steam is directed upon the bottom of the still and that it is evenly distributed throughout the oil.[1] Since the steam serves in this way to agitate it, the oil is prevented from becoming injured through overheating from contact with the bottom of the still.[2] Steam distillation is especially advantageous, however, because in this process the hydrocarbons come over at temperatures below their normal boiling points; this is due to the fact that the atmospheric pressure upon the mixture is divided between the hydrocarbons and the steam,

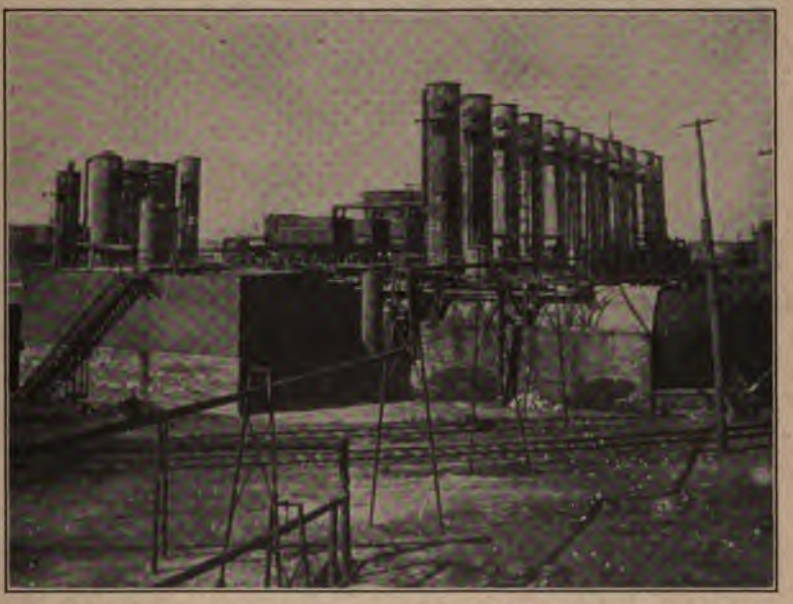


FIG. 163.—Steam stills at the plant of the Atlantic Refining Company, Philadelphia, Pa.

and the partial pressure on the hydrocarbons is less than the atmospheric pressure. The steam distillation is controlled by fires underneath the stills, the stills being well insulated other than at the heating surfaces.[3]

[1] On "steam stills," see p. 728.

[2] The steam carries the vapors away to the condenser just as rapidly as they are generated.

[3] The introduction of steam into petroleum during its distillation, was first practised in the United States by the Downer Kerosene Oil Company, at the suggestion of JOSHUA MERRILL (see p. 211); the distillation of petroleum by "high steam" was proposed by SILLIMAN in 1855 (*Am. Chemist*, **2**, 18).

An improvement in naphtha steam stills is to install over the still a high tower[1] through which the naphtha vapor from the still ascends and meets the cold, crude naphtha which flows down from the top of this tower. The heat exchange thus effected has proved a great economy in naphtha distillation. By this distillation, the crude naphtha is fractioned into a variety of products ranging from gasoline with gravities between 80° and 90°Bé. to ordinary stove gasoline with gravities between 70° and 80°Bé.



FIG. 164.—A view of the stills at the Lockport works of the Texas Company.

Motor naphthas with gravities of from 56° to 60°Bé. and certain special heavier products designed as solvents are prepared from a certain variety of crudes, especially in Texas and California.

The *vacuum process of distillation* of heavier products is in use in some American refineries, in conjunction with the steam distillation. In this process, by the aid of a pump,[2] a partial vacuum is created in the still, and, as a result thereof, the hydrocarbons

[1] See p. 731 For a separating chamber arranged and combined with a still and condenser in such a manner as to gradually separate and condense the heavier parts of the vapors formed and continually return them to the still for a further action of the heat, see LUTHER ATWOOD'S United States Patent 28246, May 15, **1860.**

[2] For an apparatus in which it is claimed that distillation can be conducted at reduced pressure without the use of an exhauster, see WANKLYN and COOPER'S English Patent 4097 of **1893.**

come over at temperatures considerably lower than their boiling points at atmospheric pressure. In general, the stills employed in the operation of this process are of the same type as the ordinary horizontal crude stills,[1] but are smaller, heavier and strongly braced.

It has long been known that there is an improvement over the results of the ordinary methods, both in the quantity and quality of the distillation products, when vacuum distillation combined with steam distillation is used.[2] However, many American refiners who are familiar with the results which can be obtained by the vacuum method, state that the difference is not great enough to warrant the increased cost of installation and operation.

*Pressure distillation*, which was, for a long time, avoided because of the hazards involved, has lately come into practical use in the production of gasoline from gas and fuel oils. The processes of "cracking," wherein employment is made of pressure and high temperatures, which may be accompanied by hydrogenation and the use of catalysts, will be considered at some length on pages 554 to 579.

On account of the importance of the subject and the detailed account required, a special section is devoted to the *chemical treatment* ("treating") of petroleum distillates in the following chapter,[3] which is, in fact, an appendix to this chapter. Brief accounts of the procedures employed in practice will, however, be given below.

**The Dry- or "Cracking"-Distillation of Paraffin-Base Petroleum.**—In the destructive distillation of paraffin-base petroleum, the condensate begins flowing into the receiving house at about 80°Bé. gravity, and the first cut is usually made from the beginning of "stream" running until the gravity of the condensate reaches 69°Bé. This product is usually termed *light naphtha* (1). The next collection is made from 69° to 58°Bé., which condensate is known as *heavy naphtha* (2). It is a general practice to steam-still this fraction into light naphtha (1) and

[1] See p. 717. For two recent designs in vacuum stills, see SCHLIEMANN, German Patent 227179 of **1909**; and STEINSCHNEIDER, United States Patent 981953 of **1911**.

[2] See GRÄFE, *Petroleum*, **3**, 1128. GRÄFE supplies a physico-chemical explanation of his results. Cf. GURWITSCH, *idem*, **4**, 265 and 618; and GRÄFE, *idem*, **4**, 268 and 621. On the advantages of high vacuum distillation, see SINGER, *idem*, **10** (1915), 605.

[3] See pp. 579 to 609.

*low-test burning oil stock* (3).[1] The light naphtha (1) is in some instances redistilled,[2] but is more frequently marketed, either without other treatment or after chemical refining,[3] as *deodorized naphtha* (4), or "benzine-naphtha." The lines of demarcation are purely arbitrary.

The third fraction, which is generally termed *high-test burning oil* (5), is collected from 58° to about 43°Bé. It is steam-stilled to *150°F. fire-test water-white burning oil distillate* (6), the condensate being combined with the heavy naphtha (2).

The next cut is made when the high-test burning oil (5) commences to go "off color." This distillate, the fourth fraction, which is known as *low-test burning oil* (7), is collected until the residue in the still reaches 21° to 22°Bé.; it is also redistilled in steam stills, the condensate being *gas naphtha* (8), a fraction of 70°Bé. gravity,[4] and the residue constituting *110° to 115°F. burning oil stock* (9). The burning oil stocks (6) and (9) are also termed "kerosene distillates."

The chemical refining of the burning oil stocks (3), (6), and (9), to remove their color and unpleasant odor, is carried out along the following general lines: The oil is agitated by means of an air blast with 5 to 10 lb. of 66°Bé. commercial sulphuric acid per barrel, the treatment being continued for from 30 to 60 min. After settling for about 4 hr., the "sludge acid"[5] is drawn off, and the oil is washed with water, with a 4° to 10°Bé. solution of caustic soda, and with water until neutral,[6] then

[1] While the term "illuminating oil" is preferable to either "burning oil" or "kerosene," the customary trade designation is employed in this section.

[2] Into *cymogene* (see p. 863), *rhigolene* (see p. 907), *canadol* (see p. 857), and *petroleum spirit*, or *benzoline* (see pp. 900 and 852). For European practice in the distillation of benzine, see WILD, *Petroleum*, **8** (1913), 668; and KESSLER, *Z. angew. Chem.*, **23** (1910), 1697.

[3] With 2 to 5 lb. of 66°Bé. commercial sulphuric acid per 50 gal., in agitators (see p. 579), followed by washing with water, agitation with a 4° to 10°Bé. gravity solution of sodium hydroxide (to make alkaline), a final washing with water (to render neutral), and settling in tanks. At one time a final redistillation from an alkali solution was made.

[4] "Gas naphtha" is clearly a result of "cracking." It is used in the cheap grades of naphtha (1).

[5] See p. 584.

[6] It has been the practice at some Pennsylvania refineries to add the sulphuric acid in three portions, a very small quantity being first used to remove the mechanically suspended water from the oil, and the second and third portions being allowed each to act for about 45 min., and to settle for about

separated and settled until bright. If the stocks contain considerable sulphur, as is the case of those from Lima (Ohio), Texas, and Canada petroleums, a special desulphurizing treatment is necessary; this matter is considered in the following chapter.[1]

The residuum remaining after light naphtha (1), heavy naphtha (2), high-test burning oil (5), and low-test burning oil (7), have been run off, is commonly referred to as *tar* (10); it possesses a gravity of from 21° to 22°Bé. and contains the paraffin oils and paraffin wax. This tar is subjected to destructive distillation to dryness in horizontal stills of 250 to 500 bbl. capacity; *tar distillate*, or *paraffin distillate* (11), and *wax tailings*[2] (12) pass over, while the *coke*[3] (13) produced by the destructive distillation remains behind. In this distillation the water in the condenser tanks[4] is kept sufficiently warm to prevent solidification of the paraffin in the distillate.

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an hour before being drawn off. To remove the acid still remaining in the oil, a shower of water issuing in fine streams from a perforated pipe at the upper part of the agitator is allowed to fall through the oil, without agitation, for about 4 or 5 hr. A final washing is then effected with agitation by the aid of the air blast. The oil is lastly agitated with 1 per cent. of a solution of caustic soda (12°Bé.), this operation being sometimes followed by washing with water. It is then "sprayed," if necessary, and run into shallow tanks for exposure to light.

The time occupied in this chemical treatment has been given as follows:

| | |
|---|---|
| Agitation with first portion of acid | 1½ hr. |
| Deposition of acid | ¾ hr. |
| Agitation with remainder of acid | 1½ hr. |
| Deposition of acid | 2 hr. |
| Washing | 1½ hr. |
| Agitation with alkali | 1½ hr. |
| Final washing | 1 hr. |
| Total | 9¾ hr. |

[1] See p. 609.

[2] This product comes over immediately prior to coking. It contains compounds produced by the "cracking" process—among these are anthracene and chrysene (see p. 858)—and is of a semi-asphaltic nature.

[3] This coke is much like gas-coke in appearance, but is more fragile; it is used for making battery and arc-light carbons, and in certain metallurgical operations. The yield of coke from "tar" amounts to approximately 10 per cent., that is, 1 per cent. from the petroleum dry-distilled. It is usually removed from the still by tools, such as picks.

[4] See p. 736. On the production of petroleum distillates of heavy gravity suitable for lubricating purposes and of high fire test and boiling point

The tar distillate (11) is the source of the paraffin lubricating oils as well as the paraffin wax; it has a gravity of 30°Bé. and a solidifying point of about 70°F. Following treatment with sulphuric acid,[1] it is delivered to the pressing plant, where the paraffin wax is separated, as described in the section on *The Manufacture of Paraffin Wax.*[2]

Formerly, the best practice was to cool the distillate in bulk, for a period of about 26 hr., in a cellular tank of 3,000 gal. capacity, fitted with oil cells alternating with cells through which circulated a solution of magnesium chloride, cooled by ammonia refrigeration. The resulting solid or crystalline mass was cautiously pressed in canvas bags by hydraulic pressure at a temperature of 25° to 30°F., and the cakes of paraffin wax thus obtained were broken up, melted, recrystallized by cooling in a similar apparatus with water, and again pressed, this time at a temperature of 70°F. and a pressure of 200 lb. per square inch. The paraffin "scale," or crude wax, thus produced amounted to about 9 per cent. of the "tar." It had a melting point of about 125°F. when obtained from oil of specific gravity 0.905, and a melting point of 117°F. when obtained from 0.885 oil. It was finally purified by crystallization from naphtha, pressing and filtration through bone-black.

The modern practice is to cool the distillate by passing it through a chilling machine, such as is described on page 756; to pass the semi-solid mass through filter presses; and to complete this separation by the process of "sweating," or fractional fusion, which is described on pages 479 and 763. The decolorization of paraffin wax by treatment with bone-black is presented on page 767.

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suitable for illuminating purposes, both being substantially free from the characteristic and offensive odor usually accompanying the heavier products of the fire-distillation of petroleum, see WILLIAM ATWOOD'S United States Patent 226151, Apr. 6, 1880. The feature of ATWOOD'S process is the condensation of the vapor of heavy-gravity distillates at a temperature above that at which the lighter gravity oil and odorous vapors will condense, so as to effect the desired separation, not by the regulation of the degree of heat employed in the vaporization, but by the regulation of the degree of heat used in the condensation.

[1] This treatment is carried out in much the same manner as the chemical refining of the burning oil stocks, but the agitator is generally heated with a steam jacket in order to maintain the tar distillate in a liquid condition.

[2] See p. 478.

The *pressed oil, pressed distillate, pressed paraffin distillate,* or *pressed tar distillate* (14), the oil expressed from the tar distillate during the separation of "slack wax,"[1] is used for the production of the various *paraffin lubricating oils,* by putting it through the process of fractional distillation with the aid of steam. Since "cracking" must be avoided, it is "steam-reduced" in reducing stills,[2] by firing underneath and simultaneously introducing steam into the oil by means of a perforated pipe coil placed inside near the bottom of the still.[3] The steam-reduction process, sometimes termed "fractional distillation," is carried out in accordance with the test of oils desired: when high-viscosity oils are being produced, the distillation is carried further than when low-viscosity oils are made; for the viscosity increases with the boiling point, since the hydrocarbons are in the same homologous series.

The first fraction in the reduction of pressed oil (15) is usually separated at 36°Bé.; it is redistilled for combining with the low-test burning oil (7). The second fraction, *fuel oil* (16), is generally cut at 32.5°Bé. The third fraction, *low-viscosity lubricating oil* (17), is almost always cut at 28°Bé.; it is redistilled by the aid of steam for the production of finished low-viscosity lubricating oil. The fourth fraction (18) is used for making *medium lubricating oil.* The residue of *heavy lubricating oil stock* or *paraffin oil stock* (19) is pumped through a cooler[4] to an agitator of from 200 to 1,000 bbl. capacity, where it is treated with from 20 to 50 lb. of 66°Bé. commercial sulphuric acid per barrel, in the same manner as described under burning oil stocks, except that the agitation is continued for from one

[1] See p. 479.

[2] These are horizontal stills of usually 300 to 500 bbl. capacity; the upper halves are bricked over. See p. 723.

[3] The "steam-reducing" is conducted along the following lines: A still is charged with the pressed paraffin distillate, and the fires are lighted. The distillation begins at about 400°F., and, shortly after this point has been reached, the live steam is injected into the oil. No water accumulates in the still because of the high temperature obtaining therein, but the steam passes upward through the oil as a gas, to be condensed with the oil vapors. Without the use of steam, the distillation would require that the still be heated to about 750°F. Such a temperature would result in obtaining an oil below the desired standard; but, with steam, the distillation can be carried on with a maximum temperature of about 600°F., thus entirely avoiding destructive distillation.

[4] This is usually a coil of cast-iron pipe set in water.



FIG. 165.—Agitators.

to two hours, and it requires a long time for the sludge acid to settle out—generally from 4 to 6 hr. The amount of sulphuric acid used in this treatment is dependent upon the color desired for the product.

After the "sludge acid" has settled and has been drawn from the base of the agitator,[1] the oil is run into a wash agitator or wash tank, in which it is agitated with a solution of caustic soda (1° to 6°Bé.),[2] then sprayed with water until neutral. The oil is finally separated from water, transferred to shallow tanks, heated to 150° to 160°F. by closed steam coils, and air-blown until clear, bright and free from water.

THE AVERAGE YIELD OF PRODUCTS BY DRY-DISTILLATION

(Paraffin-base Petroleum)

| | |
|---|---|
| Light naphtha..... | 12 to 15 per cent. |
| Heavy naphtha[3]..... | |
| Burning oil stocks[4]..... | 65 to 75 per cent. |
| Tar[5]..... | 10 to 12 per cent. |
| Loss..... | 5 to 6 per cent. |

**The Dry- or "Cracking"-Distillation of Mid-Continent Petroleum.**[6]—When the temperature of the oil in the crude still has reached 175° to 200°F., some gases, consisting largely of butane and pentane, are given off, and presently the lightest naphtha starts to distil over. The firing is continued; the temperature in the still becomes gradually higher; and the distillate becomes gradually heavier until the interior of the still reaches a temperature of about 325°F., at which point from 6 per cent. to

[1] On the recovery of sulphuric acid from sludge, see pp. 584 to 591.

[2] The "lye" which separates and is drawn off after this treatment, contains various sulpho-compounds, formed by the treatment with sulphuric acid.

On its subsequent treatment, see p. 592.

[3] The term *gasoline* (see p. 870) is applied to both light and heavy naphthas. On this point, see HYDE, *J. Ind. Eng. Chem.*, **1**, 377.

[4] The quantity of naphtha and burning oil obtained varies according to the nature of the petroleum. The amount of *water-white kerosene* obtainable is said to average 16 per cent. Cf. p. 474. For an account of the grades of illuminating oils, see p. 876.

[5] On the products of tar, see *paraffin oils*, p. 894; *paraffin wax*, p. 894; *wax tailings*, p. 921; and *coke*, p. 859.

[6] Based upon the description by F. C. ROBINSON, chief chemist of the Atlantic Refining Company, published in *Proc. Eng. Club Phila.*, April, **1913.**

8 per cent. of crude naphtha (200°F. boiling point) has distilled over. This is set aside as *crude naphtha.*

The distillation is continued until the temperature in the still has reached about 475°F., for the production of *crude heavy naphtha,* which represents 13 per cent. to 15 per cent. of the crude and has an average boiling point of about 300°F. The distillation is then carried on until the temperature in the still has reached about 625°F., for *natural lamp distillate,* which represents from 16 per cent. to 18 per cent. of the crude petroleum and has an average boiling point of about 450°F.

When the still has reached the temperature of about 625°F., "cracking," or destructive distillation, sets in. The fires are slackened in order to distil very slowly, and this slow distillation is continued until the temperature in the still reaches 675° to 700°F., producing a distillate with an average boiling point of about 550°F., but containing some gasoline, some lamp oil and much heavier oil; this may be referred to as *gas and fuel oil stock.* The yield of this oil is about 20 per cent.

The residuum in the still is a heavy black tar, representing about 42 per cent. of the crude oil. This tar is the source of paraffin wax and the line of lubricating oils called *paraffin oils.* It is no longer desirable to carry on a cracking distillation because this would result in the destruction of the valuable products. The distillation is continued in such a way as to avoid cracking as much as possible (that is, it is distilled fast), either in the same still or more commonly in separate smaller stills, called tar stills.

This tar-still distillation is carried on very rapidly in order to produce the maximum yield of *paraffin distillate* (about 22 per cent.). In addition to the paraffin distillate, there is also produced, by destructive distillation, about 15 per cent. of *cracked distillate.* At the end of the distillation the stream becomes very heavy and is then known as *wax tailings,* which amounts to about 1 per cent. of the crude oil. The *coke* which remains in the still upon the completion of the distillation amounts to about 4 per cent. of the crude oil. The paraffin distillate is delivered to the pressing plant, where paraffin wax is separated as described on page 478, and the pressed paraffin distillate is put through the process outlined on page 468.

The crude naphtha is again distilled; first, in order to steparea it into the various gasolines and naphthas that compose it, and,

secondly, to separate it from the small amount of "bottoms," or light lamp oil, that it contains. This distillation is carried out in a still which is heated by steam, usually by injecting live steam directly into the gasoline. When the distillation starts, some gas is given off, then the lightest distillate appears at the trap, usually about 90° Bé. gravity. The distillate gradually gets heavier until all the gasoline has distilled off. The receiver is then changed and the naphtha distillate is separated. At this point, about 90 per cent. has distilled off, leaving a bottom of about 10 per cent. This bottom is essentially lamp oil and is used as such.

The heavy crude naphtha is handled in the same manner, except that it contains little or no gasoline, but about 50 per cent. of bottom, or lamp oil. The cracked distillate is also distilled with steam to remove about 4 per cent. of crude naphtha.

In practice, the naphtha distillates are agitated with about 0.5 per cent. by volume and the lamp oil distillates with about 1.5 per cent. by volume of sulphuric acid, for about ½ hr. The acid sludge is drawn off, and the oil is washed with water and alkali to remove all traces of acid, and is then ready for the market.

THE AVERAGE YIELD OF PRODUCTS BY DRY-DISTILLATION
(Mid-Continent Petroleum)

Fire-stilling

| | Yield on crude oil |
|---|---|
| Crude naphtha (200°F. b.p.) | 6 to 8 per cent. |
| Crude heavy naphtha (300°F. b.p.) | 13 to 15 per cent. |
| Natural lamp distillate (450°F. b.p.) | 16 to 18 per cent. |
| Gas and fuel oil stock | 20 per cent. |
| Tar | 42 per cent. |

Tar-still distillation

| | |
|---|---|
| Paraffin distillate | 22 per cent. |
| Cracked distillate | 15 per cent. |
| Wax tailings | 1 per cent. |
| Coke | 4 per cent. |

**The Fractional Distillation of Petroleum with the Aid of Steam.**—In refineries engaged in the manufacture of high-grade lubricating oils from paraffin-base petroleum, the distillation of the latter is carried out from the beginning with the aid of dry steam, which is introduced into the oil for the minimization of decomposition. Smaller yields of gasolines, naphthas and illuminating oils and a higher yield of lubricating oils are obtained.

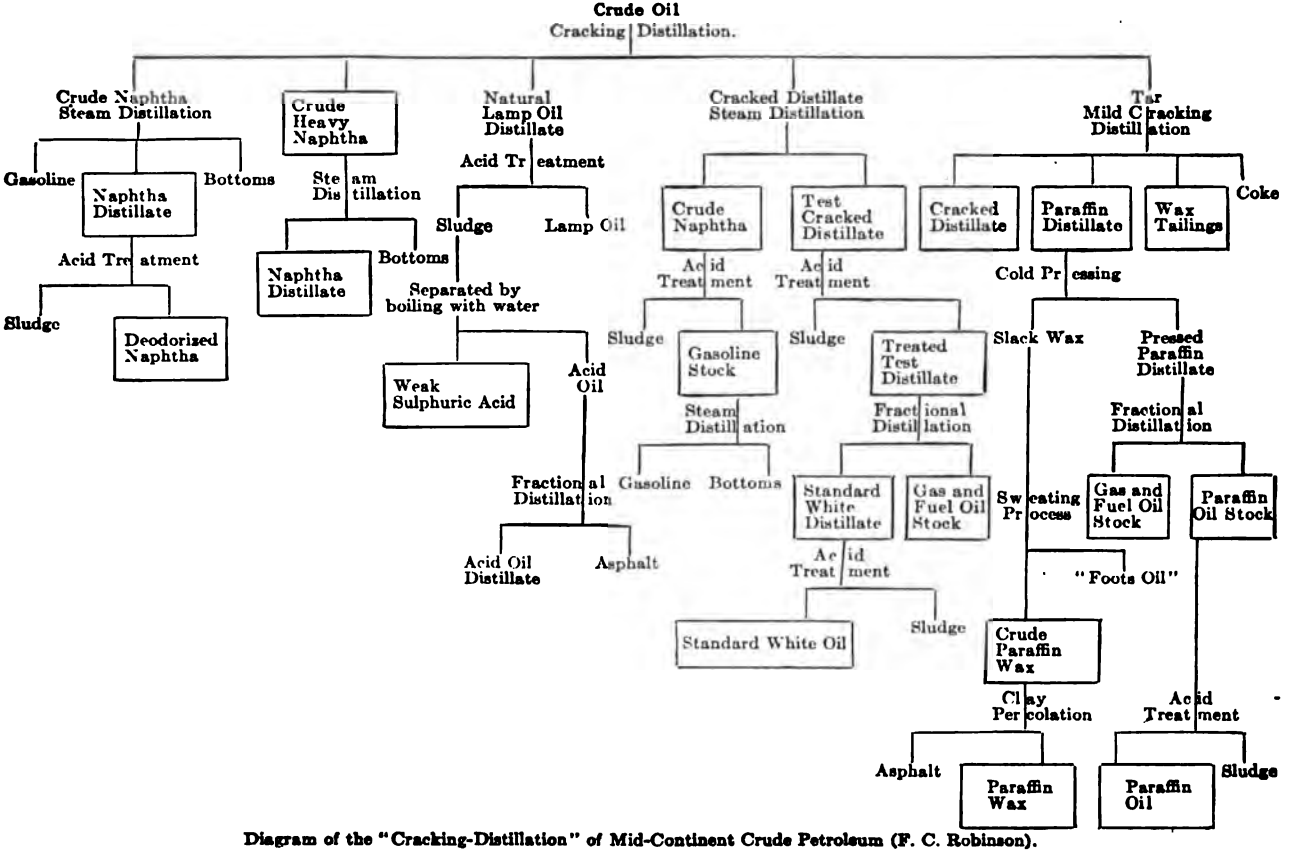



**Diagram of the "Cracking-Distillation" of Mid-Continent Crude Petroleum (F. C. Robinson).**

The residuum, or reduced stock, is termed "steam-refined cylinder stock."[1]

In practice, the lubricating crude still is charged with the crude oil, the fires are started, and the crude naphtha is distilled off as in the "cracking-distillation;" but when the temperature is well above the boiling point of water, steam is injected into the oil. Under these conditions, the crude naphtha is distilled off when the temperature in the steam still is approximately 280°F., while, without steam, the temperature in the still would be about 375°F. The yield from light-colored non-asphaltic crudes is about 13 per cent.[2] The heating is continued, and more and more steam is introduced, until the heavy crude naphtha has distilled off; at this point, the temperature in the still is about 330°F., whereas without steam it would be about 475°F. The yield of this distillate is about 13 per cent.

The fractional distillation is then continued for the production of the natural kerosene or lamp oil distillate; at this point, the temperature of the distillate in the still is about 500°F., while without steam it would approximate 630°F. At this stage of the refining, the amount of steam is increased and the distillation is conducted as rapidly as possible in order to avoid cracking (destructive distillation). This procedure is followed until the wax distillate, or lubricating distillate, has distilled over; at this point, the temperature in the still has reached about 620°F., and the fractionation is stopped, leaving the cylinder oil stock in the still. The object of distillation with bottom steam is to produce the best quality and largest yield of this stock, which is the source of the steam cylinder oils.

The crude naphtha and the crude heavy naphtha are fractioned, and certain of the distillates receive chemical treatment: the kerosene or lamp oil distillate is treated as described above; and the wax or lubricating distillate is subjected to the same process as paraffin oil, with the exception that the resulting lubricating stocks are not usually chemically treated, but are percolated through Florida earth or bone-black to obtain the

[1] On the products of the steam distillation of paraffin-base petroleums, see gasoline, p. 870; illuminating oils, p. 876; spindle oils, p. 913; cylinder stocks, p. 862; petrolatum, p. 631; turpentine substitutes, p. 918; and black oils, p. 854. See especially, however, p. 481, whereon is given a list of the products manufactured in the Appalachian field.

[2] Experience of the Atlantic Refining Company. See Robinson, *loc. cit.*

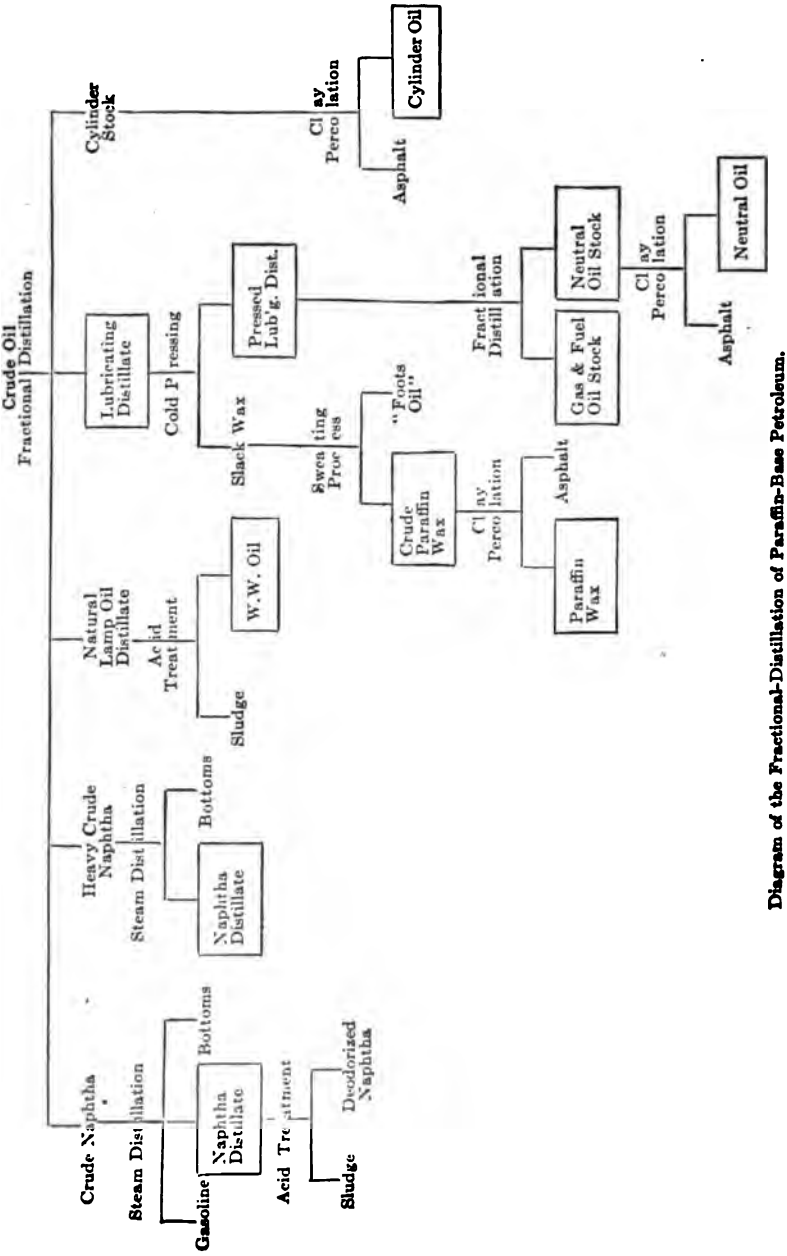



Diagram of the Fractional-Distillation of Paraffin-Base Petroleum.

neutral spindle oils. The cylinder stock is either marketed as unfiltered cylinder oil or it is run through Florida earth ("clay") to produce the filtered oils of commerce.

When a paraffin-asphalt- or mixed-base crude petroleum is put through this process of fractional distillation, the residue which corresponds to the cylinder stock from a paraffin-base crude, consists of a soft pitch which amounts to 10 to 15 per cent. of the crude petroleum. This residue is either air-blown or treated with sulphur for the production of a hard pitch which is used as a road binder, etc.[1]

**The Operations in Some Pennsylvania Refineries.**—The following brief description of the operations in the refining of paraffin-base petroleum, represents the practice at the eleven small refineries in and about Warren, Pa., visited by the authors during 1915.

The petroleum is delivered at the refineries into storage tanks[2] of a capacity varying from 10,000 to 55,000 bbl. from the pipe lines[3] or tank cars.[4]

The successive operations carried on in the refining of paraffin-base petroleum of 43° to 44°Bé. gravity, may be thus described:

The crude oil may be first steam-stilled for the production of 12 to 15 per cent. by volume of crude benzine;[5] but it is also the practice to fire-still for the recovery of this distillate, when the yield amounts to 20 to 22 per cent. The operation of steam-stilling, which gives a "sweeter" product, is conducted in horizontal stills having a capacity of 500 to 1,500 bbl., although some very small refiners use 225-bbl. stills at this stage. Fire-stilling is generally carried out in horizontal stills of a capacity of 300 to 1,000 bbl., but some refiners prefer a cheese-box still having a capacity of 150 to 1,000 bbl.[6] The stills are usually charged four-fifths full, either by pumping or, in cases where the layout permits, by gravity from the storage tanks.

The *crude benzine* is generally of 63°Bé. gravity. It is chem-

[1] See p. 626.

[2] See p. 697.

[3] See p. 249.

[4] From the tank cars the petroleum is sometimes run first into a receiving tank, through a pipe laid between the rails and equipped with suitable connections, and is then pumped into the storage tanks.

[5] This term is the one most employed by the Warren, Pa., refiners. Cf. p. 469.

[6] On the types of stills, see p. 716.



FIG. 166.—A Pennsylvania refinery which has a capacity of 35,000 bbl. of petroleum per month.

ically refined in the following manner: After transferring to the agitators,[1] which are usually of 400 bbl. capacity in the smaller Pennsylvania refineries, it is agitated by air with 5 lb. of 66°Bé. commercial sulphuric acid per barrel of distillate treated; and after active agitation for 1½ hr. and then allowing the acid-tar to settle, the sludge acid is run off and the benzine is washed by spraying cold water from the top of the agitator. This washing assists in freeing the benzine from sulphuric acid, and about as much water is used as benzine washed; the water is run off as rapidly as it accumulates in the agitator bottom. The benzine is next agitated with a solution of caustic soda (5° to 10°Bé.), which operation is a duplication of the treatment with sulphuric acid, the caustic soda and tar being run off, and the benzine washed as before.

Following the chemical treatment, the benzine is pumped or charged into the steam stills for fractionation. The condensates of this fractionation usually vary in gravity from 76° to 54°Bé.: all the condensate from the beginning of the distillation, at 80°Bé. or higher, down to 58°Bé. goes into the grades of *gasoline*, while the fraction having the gravity 58° to 54°Bé. constitutes the *deodorized naphtha*. The stills are shut down when the gravity of the condensates reaches 54°Bé., and the "bottoms" are used for working up into *kerosene*. The loss of benzine during all of the operations just mentioned amounts to from 2 to 4 per cent. The best practice is to treat the benzine continuously, whereby a large saving is effected.

In the production of *illuminating oil*, or *kerosene*, the cut is from 54° to 38°Bé., and the yield of crude kerosene amounts to about 42 per cent. This is sometimes redistilled before receiving chemical treatment. The latter is practically identical with that received by the crude benzine, except that from two or three times as much wash water is used.

Following this refining, the kerosene is run from the agitators into settling tanks or bleachers,[2] preferably of sufficient diameter to make a shallow depth; but, as an intermediate step, some refiners agitate the chemically treated oil with 1 lb. of pulverized Florida earth per barrel of kerosene, after washing with water.

[1] See p. 744 for a detailed description of the types of agitators in use. On the chemical treatment of petroleum distillates, see p. 579; cf. pp. 460 and 467.

[2] See p. 660.

The refined kerosene is left in the bleachers for from 12 to 24 hr., after which it is ready for marketing.

When a *kerosene of 150°F. fire test* is desired, the distillate cut is made from 54° to 41°Bé. *Mineral colza oil* is the cut from 41° to 38°Bé. Both condensates receive chemical treatment, but mineral colza is usually given longer agitation than crude kerosene.[1]

The distillate collected from 38° to 36.5°Bé. constitutes *fuel or gas oil*, but some refineries market the "bottoms" from the kerosene redistillation as gas oil. This product is neither chemically refined nor redistilled.

*Wax distillate* is the final condensate. It is run off from 35° to 33°Bé., and is worked up into paraffin wax and neutral oils after "cracking," as described below.[2]

The residuum, *steam-refined cylinder stock*, is pumped out from the stills through a cooler and filtered through Florida earth ("filtered cylinder oils"). It does not ordinarily receive any chemical treatment. About one-third of the petroleum refined goes into wax distillate and cylinder stock, that is, into lubricating products and paraffin wax.

The cost of manufacturing products from petroleum, in small Pennsylvania refineries, according to the above procedure, amounts to from $0.01 to $0.0125 per gallon, based on the crude oil put through the refinery.

YIELD OF PRODUCTS FROM PARAFFIN-BASE PETROLEUM

(Warren, Pa., Practice)

| | | |
|---|---|---|
| Gasoline products | | 22.0 to 25.0 per cent. |
| Illuminating oils | 150°F. fire test | 35.0 to 43.0 per cent. |
| | 300°F. fire test | 0.2 to 7.0 per cent. |
| Wax distillate[3] | | 16.0 to 18.0 per cent. |
| Cylinder stock | | 14.0 per cent. |
| Loss | | 4.0 per cent.[4] |

[1] On mineral colza oil, see p. 885. The product termed mineral seal oil, a dense oil of 39°Bé., used for lighthouse and railroad lights, was formerly made as follows: Any crude distillate, from 40° to 32°Bé., was first treated with 4 oz. of sulphuric acid to the gallon, washed with a solution of caustic soda, and distilled over soda lye. The oil had a fire test of 300°F. and but little odor. The portion of the distillate which was lower than 34°Bé. in gravity, went into the machinery oil.

[2] See p. 478.

[3] Ten per cent. of this goes into paraffin wax.

[4] Four per cent. loss is considered to be good practice.

The following account of the processes of treating Pennsylvania petroleum for the purpose of working up the whole of the products, presents in outline the refinery operations of several important companies in the Appalachian field.[1]

These companies state that while it is, of course, possible to distil the various grades of gasoline and naphtha directly from the petroleum, such a procedure does not give sufficiently "clean" products. Accordingly, the gasolines and naphthas are distilled from the benzine after it has been run off from the petroleum.

The *benzine* is divided as it comes from the condensers into two grades, termed *light* (68° to 70°Bé.) and *heavy* (60° to 63°Bé.). These products are then placed in gasoline stills, heated by steam, and fractionated into the various *gasolines and naphthas*, which are afterward refined with sulphuric acid and caustic soda in agitators.

The *illuminating oils*, usually of two grades, are distilled from the crude oil after the light and heavy benzines have been taken off. They are raised to the required fire test by expelling the more volatile portions in steam stills, but the best illuminating oils are rerun in the fire stills before being steam-stilled. On coming from the steam stills, the oil is treated with sulphuric acid and caustic soda, and is then filtered through fullers' earth. The finished products usually range in gravity from 45° to 49°Bé.

The *neutral oils*, or "spindle distillates," which carry the paraffin, are distilled from the oil after the second-grade illuminating oil has been run off. Care is exercised in cutting the distillates, so that the paraffin distils over in the neutral oils, that no paraffin comes off in the illuminating oils, and that a minimum amount of it remains in the cylinder stocks to affect the cold test thereof. All stocks contain some paraffin.

The neutral oil carrying paraffin ("spindle distillate") is termed "wax distillate." It is usually "cracked," then cooled and filter-pressed, for the recovery of paraffin wax.[2] The oil coming from the presses—"pressed distillate"—is next reduced in stills, and the resulting product, *reduced spindle oil stock* or *viscous neutral oil*, is filtered through fullers' earth or bone-black, contained in cylindrical filters of 15 to 20 tons capacity,

[1] For descriptions of refinery equipment, see pp. 644 to 776.

[2] See p. 478.

to colors ranging from dark red to white. The "pressed distillate" is usually reduced to 225 viscosity red before filtration.

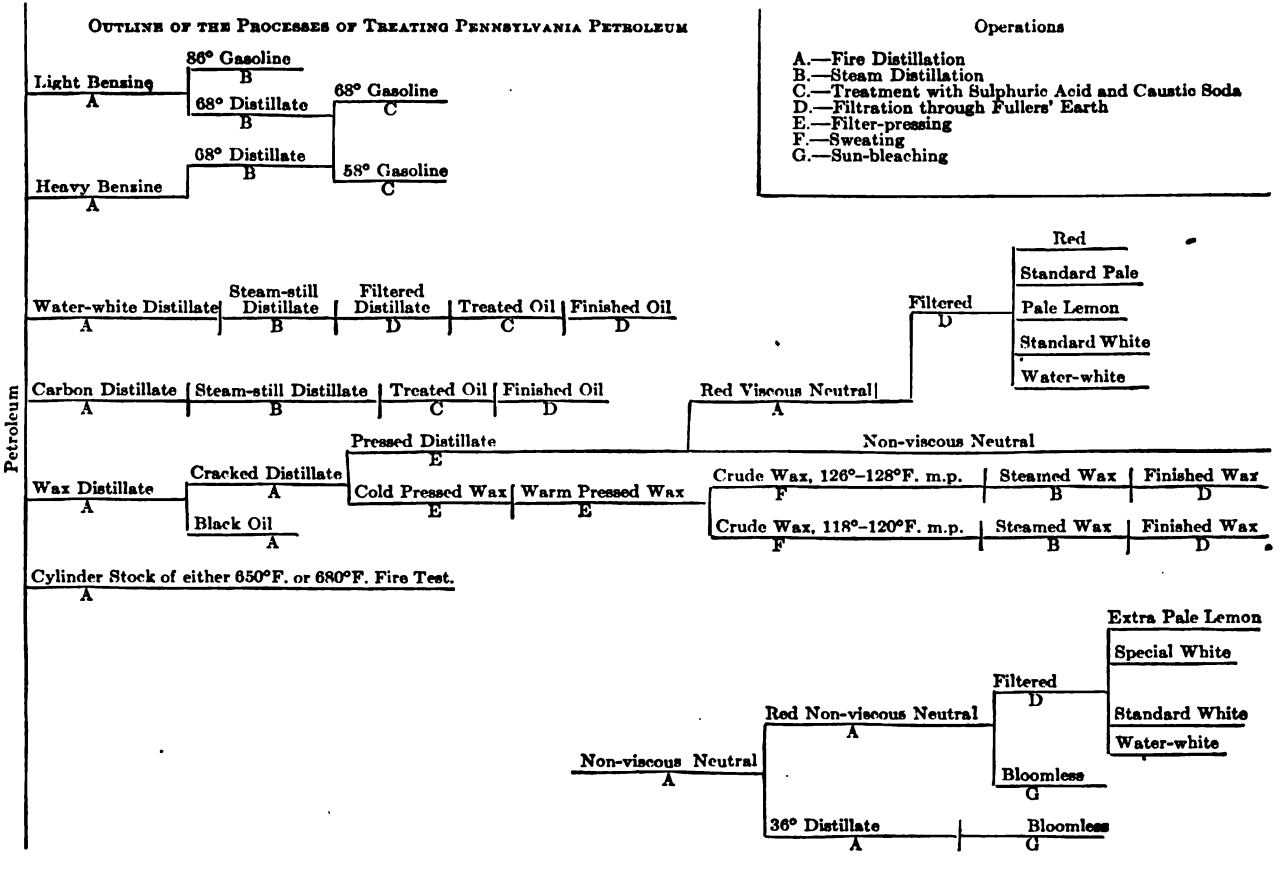
The viscous neutral oils range in viscosity from 150 to 240 on the Saybolt *A* instrument at 70°F. and the dark colored products have a gravity of from 30° to 32°Bé. It is said that the high viscosity neutrals from Pennsylvania petroleum are the nearest approach to animal oils of any petroleum products, since they are very thin and adhere tenaciously to rubbing surfaces, regardless of pressure. The claim is made that when viscous neutral oils from Pennsylvania petroleum are well over 200 Saybolt viscosity, there are no products on the market so valuable for the lubrication of engines, dynamos, automobile cylinders, compressors, or any mechanical devices requiring a high viscosity, high fire test, fluid oil, as they require no compounding whatever to perform the heaviest work that may be required.

The low viscosity or so-called "*non-viscous*" *neutral oils* are the lighter portions that distil from the "pressed distillate" in reducing that oil to viscous neutral. They are placed in stills and are slightly reduced, meanwhile "sweetening" by steam, after which they are filtered to the various colors up to white; none receives an acid treatment. Non-viscous neutrals from Pennsylvania petroleum are said to make valuable lubricants for light, high-speed work.

*Sun-bleached neutral oil* is prepared by placing the light product distilled from the non-viscous neutrals in pans 20 ft. by 40 ft., and sun-bleaching at a depth of about 8 in. over water. Sun-bleached bloomless oils are said to hold their color and to be valuable for adulterations, wool stocks, miners' oils and illuminating purposes where a high fire test is required. Neutral oils are usually sunned before filtration, to diminish the fluorescence.

*Steam-refined cylinder stock* constitutes the residue which remains in the still after the other products have been run off. These stocks vary in fire test from 500°F. to 700°F. and none over 610°F. fire test is filtered. 600°F. stocks are used to a far greater extent than any other tests. When flash, fire, viscosity and cold tests are satisfactory, the principal requirement in all stocks is that they be free from tar; high-test stocks are much more likely to contain this undesirable impurity than those of lower tests.

The *refined paraffin wax* is of two melting points, viz., 118°–


Outline of the Processes of Treating Pennsylvania Petroleum
Operations
A.—Fire Distillation
B.—Steam Distillation
C.—Treatment with Sulphuric Acid and Caustic Soda
D.—Filtration through Fullers' Earth
E.—Filter-pressing
F.—Sweating
G.—Sun-bleaching
Petroleum
Light Bensine A
86° Gasoline B
68° Distillate B
68° Gasoline C
Heavy Bensine A
68° Distillate B
58° Gasoline C
Water-white Distillate A
Steam-still Distillate B
Filtered Distillate D
Treated Oil C
Finished Oil D
Carbon Distillate A
Steam-still Distillate B
Treated Oil C
Finished Oil D
Red Viscous Neutral A
Filtered D
Red
Standard Pale
Pale Lemon
Standard White
Water-white
Pressed Distillate E
Non-viscous Neutral
Wax Distillate A
Cracked Distillate A
Black Oil A
Cold Pressed Wax E
Warm Pressed Wax E
Crude Wax, 126°–128°F. m.p. F
Steamed Wax B
Finished Wax D
Crude Wax, 118°–120°F. m.p. F
Steamed Wax B
Finished Wax D
Cylinder Stock of either 650°F. or 680°F. Fire Test. A
Non-viscous Neutral A
Red Non-viscous Neutral A
Filtered D
Extra Pale Lemon
Special White
Standard White
Water-white
Bloomless G
36° Distillate A
Bloomless G

120°F. and 126°–128°F. It is formed into cakes about 15 in. by 20 in. by 2½ in. thick; these cakes are packed in paper in cases weighing about 110 lb. net and in bags of about 120 lb. gross.

**The Manufacture of Paraffin Wax.**[1]—After the distillation has reached the stage where the boiling point exceeds 450°F., the paraffin wax and lubricating oils, called "paraffin oils," distil over. The fires are slackened in order to distil very slowly. This is continued until the temperature in the still reaches 675° to 700°F., producing a distillate of an average boiling point of 550°F. This distillate may carry some gasoline, illuminating oil, and much heavier oil, known as gas oil and "fuel oil stock," which is further distilled off.

*Preparation.*—Depending upon the nature and properties of the crude to be handled, refiners have different methods in preparing the wax distillate, from which certain lubricating oils and paraffin wax are made. A wax distillate may be rerun or "cracked" in the stills, thereby giving a distillate, crystalline in form, which affords a more complete separation of the wax from the oil in the filter presses. Some distillates, on the other hand, are not rerun in stills and satisfactory yields are obtained.

In rerunning a distillate in a lubricating still, too much steam makes a poor-pressing distillate, but it increases the yield of viscous neutrals from the pressed oil. The practices are varied and so it devolves upon the refiner to work out the best method to get the products desired. It is generally understood that a rerun distillate will always press in every way better and more easily than an original distillate from the first distillation. A distillate having poor pressing qualities, naturally requires more filter-press capacity for a given throughput of distillate.

*Process of Cold Pressing.*—The paraffin distillate obtained is is pumped into storage tanks, ready for the operations in the wax-chilling and pressing plant. The distillate is first cooled to a temperature of 15° to 30°F., depending on the cold test of the pressed oil required. This is accomplished by pumping the distillate from storage tanks through the pipes of a chilling machine designed for this purpose; the usual range is from 90°F. going in to 15°F. coming out. The chilling causes the paraffin wax (amounting to about 10 per cent. of the distillate) to solidify.

[1] This description applies to both paraffin-base and paraffin-asphalt base petroleums.

This solid 10 per cent., mixed with the 90 per cent. liquid oil, forms a soft mush which is pumped through a high-pressure hydraulic filter-press. That which remains in the press is called the "slack wax" and amounts to about 20 per cent. of the paraffin distillate. The 80 per cent. that goes through is called "pressed distillate," or "pressed oil." This chilling and pressing is carried on continuously with one pumping operation.

To produce the chilling effect or refrigeration, an ammonia absorption type of refrigerating machine is generally used, on account of its suitability for low temperatures. Calcium chloride brine of proper strength not to freeze is the medium circulated through the horizontal, double-pipe chilling machines after being cooled by the absorption machine. The brine is pumped into the bottom of the chilling machine and is circulated through the annular space between the 6-in. and 8-in. pipe thereof. The outlet is at the top. The distillate enters the inner pipe, traveling counter-current to the cold brine. The outlet is at the bottom run of pipe and thence the distillate is delivered direct to the filter presses in a chilled solid mass for separation or filtering.

The cold unpressed distillate is pumped into the press through a line provided with relief valves, set at a pressure of, say, 300 to 350 lb. gauge, to prevent too high a pressure being put on the press. The time of running a press is governed by the nature of the distillate handled. A distillate which is poorly crystallized or in which the crystals are fine, is much slower in separating the pressed oil. When the pressing is completed, the setting-up ram is reversed so as to pull back the press platen. This allows the press men to dump the "slack wax" from the spacing rings, which are generally ½ in. thick. This "slack wax" cake is usually hard and dry. It falls to a conveyor and is carried to slack wax tanks outside of the building, which are provided with steam coils for melting the wax. The "slack wax," consisting of about equal parts of oil and wax, is then put through the sweating process.

*Sweating Process.*—This process consists of cooling the mixture until it has become a solid cake and then very gradually warming it. The crystals of the paraffin form a network through which the oil is distributed and, when the mass is warmed, the oil sweats out and drips away. It always carries with it some wax in solution, but the final result is that the oil all sweats out, leaving the paraffin wax in a fairly pure state. This sweating process

separates the slack wax into crude paraffin wax and what is known as "foots oil." The latter still contains much paraffin, which is removed by putting it again through either the cold pressing or sweating process.

In sweating "slack wax" a series of pans placed one above the other in a brick building, is used. There is no particular design or size, but each refiner has his own ideas as to the construction details.[1]

It is customary to first fill these pans with cold water to a point about ⅛-in. above the level of the screens, then to pump melted wax into the pans to a depth of 4 in., after which it is cooled by means of cold water circulation through the coils in the pans. As soon as the wax is cooled or hardened to the point where it will stand up on the screens, the water under the body of wax is drawn off from the central outlet. After the water is thoroughly drained from the pans, the doors, windows and ventilators are closed and steam is turned into the heating coils, located on the walls. This then brings the room temperature up gradually to a point near or at the ultimate melting point of the sweated wax. Care should be taken not to get the room temperature too high, in which case there would be melting and breaking down of crystalline structure, instead of sweating, and thus needless loss of material. It usually requires about 24 hr. to sweat a batch of melted wax, depending largely upon how it separates.

When the sweated wax has reached the required point, as determined by making a test for percentage of oil and melting point, steam is turned into the coils in pans, thus melting down the fibrous or porous body of wax, which runs by gravity to receiving tanks below. Some prefer to have separate steam coils below the screens, instead of using the same coils for both water and steam. The melting point of wax is not a sufficient indication of the percentage of oil it may contain. For commercial purposes, the melting point varies from 118° to 126°F.

*Decolorization.*—The crude paraffin wax is finally percolated through Florida earth ("clay") or bone-black, for the purpose of eliminating tarry and asphaltic coloring matters and thereby converting it into refined colorless paraffin wax.[2]

[1] On some of the important engineering details, see p. 763.

[2] At the big eastern refineries, the "clay" is put into large upright cylinders holding from 10 to 20 tons and provided with a finely perforated bottom.

*Capacity.*—Wax plants are designed to handle a certain quantity of unpressed wax distillate. The smallest unit considered practical to run, is 100 bbl. (42 gal. each) per day of 24 hr. The larger plants are designed to handle a quantity, dependent on the amount of crude to be run daily in the stills. It is necessary to know the nature of the crude, for different percentages of wax distillate are obtained.

Information regarding the equipment of wax plants may be found on pages 753 to 772 of Chap. XIV.

The methods employed in the production and refining of paraffin wax at the Scottish shale-oil works, will be referred to in Chap. XVII.[1]

**Petroleum Products Manufactured in the Appalachian Field.**[2]—The principal products derived from the petroleum of the Appalachian field are as follows:

*Gasoline*[3] and *naphtha*[4] are made in gravities varying from 56°Bé. to 86°Bé. Gasoline used principally for automobile purposes is from 60°Bé. to 74°Bé.; the heavier grades of gasoline are used for blending with natural gas gasoline. Light naphtha is employed in the manufacture of rubber cements and in dry cleaning, while the heavier grades are used in paint mixing. Gasolines and naphthas vary in average boiling point according to the use for which they are intended, but usually lie between 125°F. and 280°F.

*Kerosenes*, also known as burning, illuminating or lamp oils, are generally made in standard grades of 47°Bé. gravity, 150°F.

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The crude wax is melted and poured on top of the "clay." It trickles down through the bed and passes through the perforated bottom. It is said that a ton of "clay" will yield 5 to 6 tons of first-quality paraffin wax.

On plant for decolorizing paraffin wax by means of bone-black, see p. 767.

On the regeneration of spent Florida earth, see p. 617.

[1] On practice in the continent of Europe, see SINGER, *Petroleum*, **8**, 1428 and 1611; PORGES, *idem*, **9**, 18; FLEISCHER, *idem*, **9**, 87; WEISER-MATA, *Z. angew. Chem.*, **33**, 2270; and ENGLER and HÖFER'S "Das Erdöl," **3**.

[2] For definitions of the available trade names of the products of petroleum, see Chap. XVIII, pp. 845 to 923. The nomenclature of petroleum products is often confusing.

[3] A good practice is to use the term gasoline for any mixture of light hydrocarbons intended for use in any kind of vaporizer, that is, to be gasified in a gas machine, gasoline torch, gasoline stove or automobile carburetor.

[4] The word naphtha is here applied to mixtures of light hydrocarbons intended for some purpose that requires a very good odor, such as varnish making, soap making, and cleaning.

fire test; 47½°Bé. gravity, 150°F. fire test; 48°Bé. gravity, 150° fire test; 49°Bé. gravity, 130°F. fire test; and export oil, 110°F. fire test.

In addition to these grades, others are made for special purposes: 300°F. fire test for railroad signals[1] and 48° gravity 150°F. fire test for incubators,[2] are instances of special illuminating oils.[3]

Illuminating oil is understood by most refiners to be a mixture of hydrocarbons whose average boiling point is about 450°F., entirely freed on the one hand from gasoline or naphtha and on the other hand from the heavy hydrocarbons that belong to gas oil and lubricating oil.

*Non-viscous neutral oils* are made in different colors and tests and are used for a great many different purposes, among which are oils for all grades and kinds of light and fast running machinery, cordage oil, wool oil, road oils, etc. Neutral oils below 150 viscosity on the Saybolt instrument are generally included in the term non-viscous oils.

*Cylinder Oils.*—Pennsylvania petroleum yields an oil for use in steam cylinders, which is made in various tests, ranging from flash test 540°F. to 630°F., equivalent to a fire test of 600°F. to 710°F. Steam cylinder oils must possess such a flash that they will not evaporate in a steam cylinder and the proper viscosity for the use intended at the temperature of the cylinder.

*Automobile Oils.*—These are used in automobile cylinders. The essential requirements for an oil for this purpose are proper viscosity and high flash test, with a minimum tendency toward carbonization.[4] The refiners of Pennsylvania petroleum claim that their neutral oils, properly refined and filtered, possess these qualifications.[5] Automobile oils are made in various tests, ranging principally from flash test 390°F. to flash test 450°F.,

[1] See mineral seal oil, p. 885.

[2] See incubator oil, p. 876.

[3] See p. 876 for other comments on illuminating oils.

[4] See spindle oil, p. 913. On the systematic oiling of all important parts of the automobile mechanism, see *Sci. Am.*, Jan. 2, 1915, 18. For a general consideration of motor cylinder lubrication, see G. S. Bryan, *J. Am. Soc. Mech. Eng.*, 37 (1915), 293.

[5] Bryan (*op. cit.*) states that oils from southern asphalt-base crudes have shown less carbon-forming proclivities in the cylinder than oils from Pennsylvania petroleum.

equivalent to fire test 440°F. to 550°F.; the viscosities vary from 150 to 240 on the Saybolt instrument at 70°F.

*Gas engine oils* are made in various tests and colors for other internal combustion engines. The requirements are similar.

*Paraffin wax* is made in grades termed by the trade "yellow crude scale," "white scale," and "refined wax." It is made to melt at temperatures ranging generally from 110°F. to 130°F., the standard grades being 118°/120° m.p., 122°/124° m.p., and 124°/126° m.p. The refined wax is pure white; it should contain less than 0.5 per cent. of oil and moisture and be practically odorless and tasteless.

The above-mentioned products are the principal ones derived from Pennsylvania crude petroleum. There are others obtained, such as petrolatum, medicinal mineral oil, and turpentine substitute, a product similar to heavy benzine; but these are produced only in limited quantities and, with the exception of petrolatum, are not of much commercial importance.

**The Operations in the Refining of Asphalt-Base Petroleum.**—The refining of asphalt-base petroleum *in toto* is substantially the same as presented in the methods of refining paraffin-base crude oil. The residue is, however, asphalt,[1] instead of cylinder stock, and, except in the case of paraffin-asphalt or semi-asphalt base petroleums, no wax distillate is obtained. On the other hand, a larger yield of fuel oil is procurable.[2]

[1] The proper distillation at low temperatures of California petroleum, in order to obtain a suitable residue, was first attempted by the Barber Asphalt Paving Company. See the following descriptive notes in Chap. XVIII.: Asphalt, Bitumen, Bituminous Materials, and "Condensed" or "Blown" Oils. Cf. the following chapter, pp. 620 to 631.

[2] The crude oil of the Kansas, Oklahoma, Louisiana, and Texas fields is refined in two types of plant—those which produce only naphtha or gasoline, illuminating oils, and, in some cases, gas oil, the residuum being sold for fuel oil; and those which, in addition to obtaining some of the above-mentioned products, also produce lubricants from the residuum. The first type of refineries is commonly known as "skimming" or "topping" plants; the second as "lubricating" plants. Most of the refineries situated west of the Mississippi River do not produce lubricants. The reasons for this are found in their comparatively limited markets for lubricating oils, in the more expensive plant and higher grade of skill needed to manufacture lubricants, and, to some extent, also in the profitable local markets for fuel oil. As a result, comparatively little lubricating oil is refined west of the Mississippi River from the crude oil of the Mid-Continent, Louisiana, and Texas fields, when the quantity of the crude oil, which unquestionably has considerable lubricant possibilities, is considered. The refiners have regarded it more

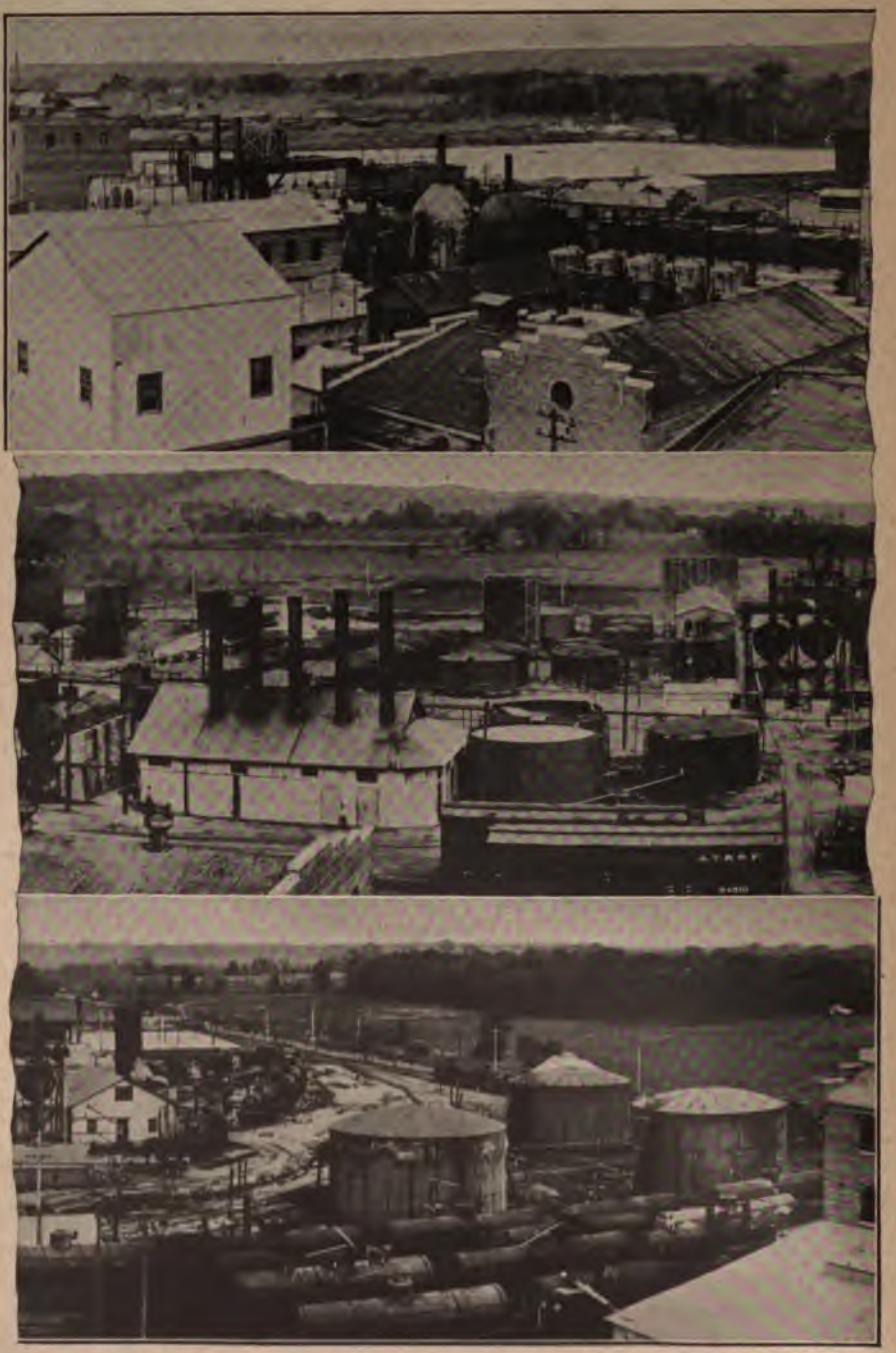


FIG. 167.—The plant of the Cudahy Refining Company at Coffeyville, Kansas. This refinery, which was erected in 1909 and covers 45 acres, has a still capacity of 5,000 bbl. per day; a general line of petroleum products is made from Oklahoma crude oil.

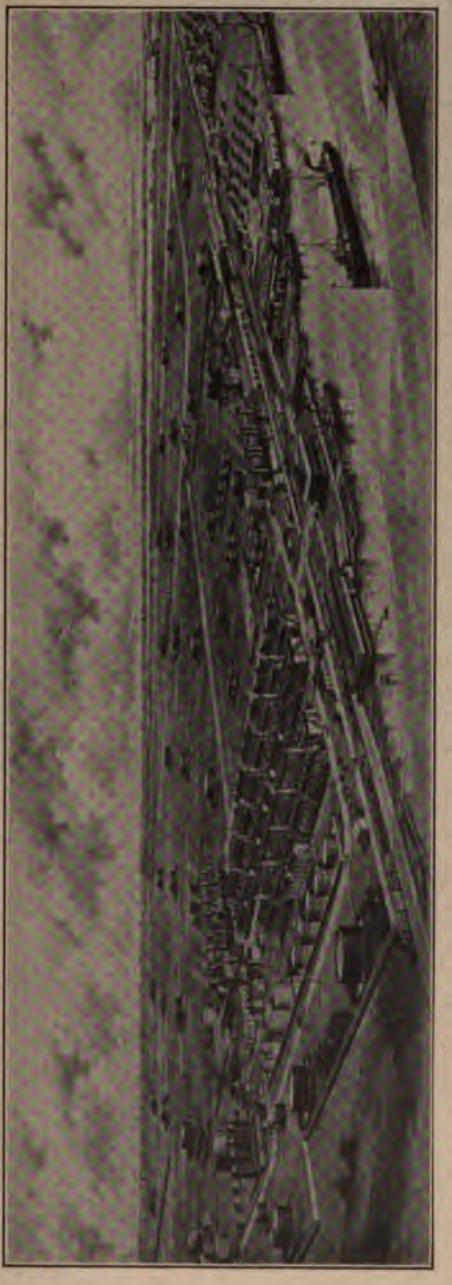


FIG. 168.—The Port Arthur, Texas, refinery of the Gulf Refining Company.

In the distillation of asphalt-base petroleums to a tar, called by some asphaltum oil or fluxing oil, the distillates are cut according to gravity, as in the case of the processes described. If, however, the crude oil is essentially a fuel oil, and it is only necessary to distil off a small amount of naphtha and burning oil in order to obtain a fuel oil which may be stored with safety, the distillation is carried on until the distillate, which is passed through a condenser and appears in the tail house, indicates by its gravity that the oil remaining in the still will have a flash-point above a degree designated as safe—usually 150°F. The oil is then pumped through a cooler into the proper tanks for distribution to the points of use.

In the manufacture of road oils, the process is much the same, although the practice is becoming more frequent of carrying the distillation until the residuum left in the still is largely asphaltic oil that is almost solid when cold. Steam is used very generally in the reduction of crude oils for road purposes, because the resulting oil is more elastic than would be the case if steam were not used. Without steam, the oil is cracked into a product which is more brittle.

Prior to 1908 the oil production in the State of California had been almost entirely a heavy fuel oil, with a high flash-point, but changed within a short period to a large percentage of refining oil with a low flash, by reason of the coming in of the Santa Maria field, the Midway field, and the increased production of the Fullerton field.

This is clearly shown in the following tabulation of California's production:

| Year | Barrels | Below 20°Bé., per cent. | Above 20°Bé., per cent. |
|---|---|---|---|
| 1908 | 48,307,000 | 90 | 10 |
| 1910 | 77,698,000 | 80 | 20 |
| 1912 | 90,075,000 | 69 | 31 |
| 1914 | 103,600,000 | 47 | 53 |

The bringing in of these high percentages of light oil with their low flash and the increasing demand for refined products led to

expedient or profitable to "top" their crude and sell the residuum for fuel oil. When market conditions warrant, sometimes part of the residuum is run to gas oil, at other times all of it goes into fuel oil.

the devising of topping plants as a cheap and efficient means of quickly separating the lighter oils, either in the field or at the main distributing points.

The plant devised by M. J. Trumble has been installed at several refineries in California, for the production of gasoline, "distillate," and fuel oil.[1] In the Trumble "evaporator," the petroleum is fed into the upper part of a chamber having vertical walls and heated externally by suitable means; it is caused to spread over the walls in a thin film by means of conical spreaders, and the vapors given off are withdrawn through openings in a



FIG. 169.—View at a recently erected California refinery, showing the 10 ft. by 40 ft. steam stills with their towers and condenser boxes, and (to the extreme left) an agitator.

central vapor pipe having horizontal discharge pipes passing through the walls of the evaporator and through the heating jacket to a collecting main without.[2] The Trumble apparatus for refining petroleum comprises a series of compartments, each supplied with an overflow to the succeeding compartment at such a height that the petroleum sinks from level to level as it passes through the compartments. The petroleum is heated by steam coils in the compartments and the distillate is condensed in a single condenser chamber extending over the top of the compartments and communicating with each one. In practice, the

[1] See p. 503 for definitions of these specific products.

[2] United States Patent 996736, July 4, 1911; French Patent 431142, June 16, 1911.

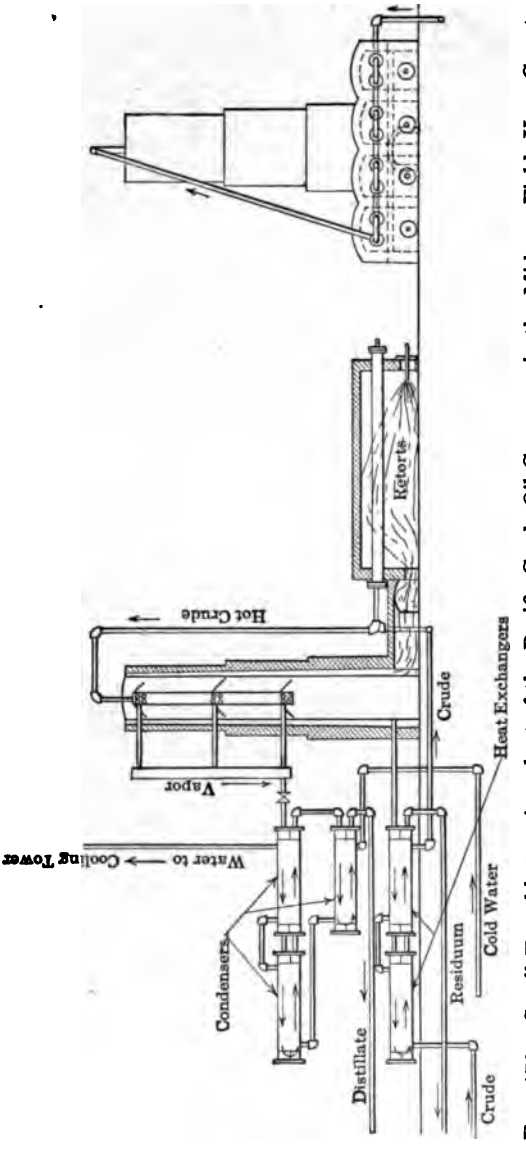



FIG. 170.—Small Trumble topping plant of the Pacific Crude Oil Company in the Midway Field, Kern County, California.

oil vapor from a fire still is condensed by passing it through a pipe having a water-jacket and the condensate is then passed into the first refining compartment.[1]

The special features claimed for the Trumble process are as follows:

The saving of heat in the evaporating chamber by means of conical spreaders in the evaporators, all oil being forced to run down the inside surface of the shell, which is heated on the outside by the escaping flue gases.

The condensing of the first-run vapors and the re-evaporation of these distillates by passing them through a specially designed separator, which derives its heat from the outflowing first-run vapors.

The recovery of the re-evaporated distillates from separate compartments of the separator, thereby furnishing finished products of different gravities.

Other topping processes and plants have been devised by H. G. Burroughs,[2] E. I. Dyer,[3] and Brown and Pickering.[3] For detailed descriptions of the important topping plants of California, the reader is referred to the paper on this subject by A. F. L. Bell in the *Bulletin of the American Institute of Mining Engineers*, **1915**, No. **105**, 1769–99.

**Petroleum Products Manufactured in the Mid-Continent Field.**[4]—The following is a description of such products as are now being manufactured in what are generally known as "stripping plants," that is, such plants as do not produce lubricating oil; this descriptive list includes such products as are made and characterized by the refiners generally in the Mid-Continent field.

*Benzine.*—The raw condensate distilled from crude petroleum, including all such condensate from the lightest fractions down to about 52°Bé. gravity.

*Gasoline.*—The finished, steam-refined product from the benzine, including the entire product from the lightest fractions down to that point which will yield a combined product of about

[1] United States Patents 1002474, Sept. 5, **1911**; and 1070361, Aug. 12, **1913**. On the TRUMBLE process, see *Petrol. World*, **1915**, 178; and *Mining Eng. World*, **43**, 811.

[2] United States Patent 998837, July 25, **1911**.

[3] See BELL, *cit. supra*.

[4] The information included in this section was secured by careful inquiry of the companies operating in the Mid-Continent field.

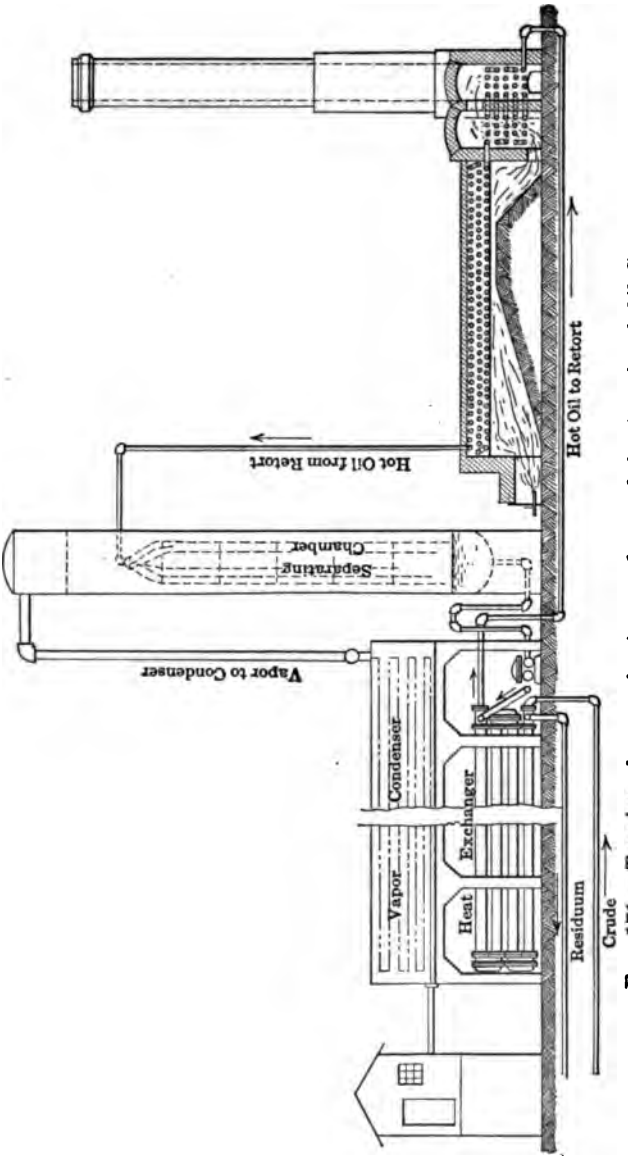



FIG. 171.—Topping plant at the Avon refinery of the Associated Oil Company.

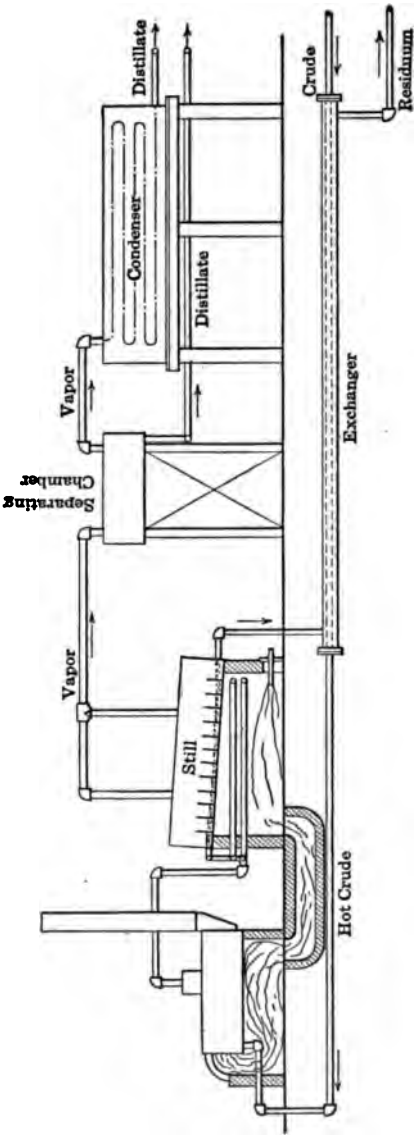



FIG. 172.—Brown-Pickering plant of the Union Oil Company, Naranjal, Fullerton Field, California.

60°Bé. gravity. Three grades of gasoline are generally produced in Kansas and Oklahoma: 68°–70°, 64°–66°, and 60°–61°Bé.; the last two grades are ordinarily put out for use in automobiles.

*Naphtha.*—That portion of the steam-refined benzine of gravity heavier than gasoline, including all the product from the highest products following those cut into gasoline down to the heaviest products which come over into the worm free from the greasy kerosene fractions, and showing a gravity of from 50° to 58°Bé.

The Coffeyville, Kansas, refineries produce the following grades of naphtha:

54°–56° *Naphtha.*—A light naphtha, or heavy gasoline, used largely in the paint trade and by dry cleaners.

50°–52° *Naphtha.*—A heavier cut, sometimes used in the place of 54°–56° naphtha.

49° *Naphtha, or Turpentine Substitute.*—This grade of naphtha is said to be largely used by varnish makers in place of turpentine.

*Kerosene* is usually cut from about the fraction showing a gravity of 52°Bé. down to that having a gravity of 36°Bé., the lowest point for good color and freedom from such fractions as would show a wax cloud at a temperature of 0°F. or viscous portions which would prevent free flow through a lamp wick. These condensates are chemically treated with sulphuric acid and caustic soda.

The following grades of kerosene are produced from Kansas petroleum:

44–46° *Kerosene.*—The highest grade kerosene, composed of the lighter portions of the kerosene distillate.

43°–44° *Kerosene.*—An intermediate grade.

40°–42° *Kerosene.*—The ordinary grade of kerosene put out in tank wagons.

*Power Distillate, Kerosene Distillate, and Petroleum Distillate.*—The untreated condensates as described under kerosene and in addition thereto still heavier distillates down to fractions as low as about 28°Bé.; these distillates (especially that of gravity 39° to 40°Bé.) are used as fuel in internal combustion engines, different grades being used in various types of engines. They are also used for gas-making purposes, the gravity of the oils being determined by the requirements of the user. A product of Kansas petroleum, termed *Gas Oil*, which is used in the manufacture of gas, has a gravity of 34° to 36°Bé.

*Fuel Oil.*—An indefinite term, properly used to describe any product of petroleum used for the production of power or heat, ranging from the distillate series down to and including any product which can be made liquid by steam heat and for which no better market outlet can be obtained. There are no fixed specifications for Mid-Continent fuel oil, each user being guided by his own peculiar needs and facilities; it must not, however, contain gasoline, and many consumers require that it be of such a consistency that it can be pumped through pipes and burners.

*Residuum (Petroleum Tailings).*—Any residue left in a still after the usual process of distillation has been completed; more properly, the residue of crude oil, rather than the residue from the redistilled condensates.

*Asphalt (Liquid Asphalt).*—The residuum from asphalt-base crude oil; it is of many grades, consistencies and melting points, according to the needs of the user.[1]

*Road Oil.*—An indefinite trade name covering grades of oil, ranging from 34°Bé. distillate, used for sprinkling playgrounds, etc., as a dust preventative, down to an asphalt, solid at 60°F., depending entirely on the needs of the user and the conditions under which it is to be applied.

The following grades of road oil are produced from Kansas petroleum: 24°–25°Bé. oil, a light grade, used largely as a dust settler; 22°–23°Bé. oil, an intermediate grade; and 20°–21°Bé. oil, a heavy road oil or binder, used on macadam and dirt roads.

One prominent Oklahoma refining company markets the following road oils:

30°–34° *Road Oil.*—This product showed a flash-point (N. Y. oil tester) of 250°F., a fire-point of 300°F., a viscosity at 60°F. (Engler) of 132 sec., and 1.1 per cent. evaporation after 5 hr. at 212°F.

26°–30° *Oil.*—An examination of this product showed a flash-point of 330°F., a fire-point of 390°F., a viscosity at 60°F. of 563 sec., and 0.2 per cent. evaporation after 5 hr. at 212°F.

24°–26° *Oil.*—This usually possesses a flash-point of 160°F., a fire-point of 220°F., a viscosity at 77°F. of 715 sec., 4.38 per cent. evaporation after 5 hr. at 212°F., 1.5 per cent. of fixed carbon,

[1] The marketed production of oil asphalt derived from petroleum of domestic origin in 1916 amounted to 688,334 short tons. The quantity of oil asphalt from Mexican petroleum manufactured wholly within the United States in 1916 was 572,387 short tons.

and an asphaltic content of 40 per cent. (100 penetration at 77°F.).

20°–22° *Oil.*—An analysis of this product gave a flash-point of 350°F., a fire-point of 400°F., a viscosity at 212°F. of 120 sec., no evaporation after 5 hr. at 212°F. and 3.38 per cent. evaporation after 5 hr. at 325°F., 2.5 per cent. of fixed carbon, and an asphaltic content of 54 per cent.

18°–20° *Oil.*—A sample of this oil showed a flash-point of 420°F., a fire-point of 530°F., a viscosity at 212°F. of 276 sec., 0.45 per cent. evaporation after 5 hr. at 325°F., 3.9 per cent. of fixed carbon, and an asphaltic content of 77 per cent.

16°–18° *Oil.*—An analysis of this product gave a flash-point of 410°F., a fire-point of 540°F., a viscosity at 212°F. of 320 sec., 0.65 per cent. evaporation after 5 hr. at 325°F., 4.4 per cent. of fixed carbon, and an asphaltic content of 81 per cent.

The same company also produces 11°–14°Bé. and 14°–16°Bé. flux oils.

The following grades of lubricating oils are produced at certain refineries:[1] *auto oil* (usually of 450°F. fire test); *gas engine oil*, a heavy red oil of 450°F. fire test; *red engine oil* (usually of 450°F. fire test); *cylinder oil* of about 600°F. fire test; *harvester oil*, a product of high fire test and viscosity which is sometimes compounded with graphite for use on heavy engines and farm machinery; *castor machine oil*, for use on harvester and farm machinery; *cream separator oil* (over 300°F. fire test); and *floor oil.*

**The Value of Oklahoma Petroleum.**[2]—Notwithstanding the fact that Oklahoma is one of the principal oil-producing areas in the world, very little work relating to the character of its oils has been reported in the literature. David T. Day, of the

[1] The following is a list of Mid-Continent refineries having lubricating plants in 1916: Great Western Oil Refining Company, Erie, Kan. Kansas City Refining Company, Kansas City, Kan. Kansas Co-operative Refining Company, Chanute, Kan. National Refining Company, Coffeyville, Kan. Cudahy Refining Company, Coffeyville, Kan. Petroleum Products Company, Independence, Kan. Muskogee Refining Company, Muskogee, Okla. American Refining Company, Okmulgee, Okla. Milliken Refining Company, Vinita, Okla. Consumers' Refining Company, Cushing, Okla. Ponca Refining Company, Ponca City, Okla. Cosden & Co., Tulsa, Okla. Magnolia Petroleum Company, Fort Worth, Tex.

[2] See BUTTRAM, *Bull.* 18, *Okla. Geol. Survey*, 1914, 52; and "Conditions in the Healdton Oil Field," Bureau of Corporations, Washington, D. C., 1915.

TABLE XLIII.—COMPARATIVE VALUE OF CUSHING, GLENN POOL, AND AVERAGE OKLAHOMA, AND ELECTRA (TEX.) AND MANSFIELD (LA.) CRUDE OILS, AS DETERMINED BY REGULAR REFINERY OPERATIONS (BASED UPON 1 BBL. OF 42 GAL. OF CRUDE)[1]

| Crude oil and product | Per cent. yield | Gallons | Price per gallon | Amount |
|---|---|---|---|---|
| Electra Crude:[a] | | | | |
| Gasoline | 22.89 | 9.61 | $0.07081 | $0.68048 |
| Engine naphtha | 2.81 | 1.18 | 0.05766 | 0.06804 |
| Kerosene | 25.87 | 10.87 | 0.04796 | 0.52133 |
| Gas oil | 9.33 | 3.92 | 0.04500 | 0.17640 |
| Fuel oil | 36.77 | 15.44 | 0.01875 | 0.28950 |
| Loss | 2.33 | 0.98 | | |
| Total | 100.00 | 42.00 | ..... | $1.73575 |
| Mansfield Crude:[b] | | | | |
| Gasoline | 10.00 | 4.20 | $0.07081 | $0.29740 |
| Kerosene | 55.00 | 23.10 | 0.04796 | 1.10788 |
| Gas oil | 20.00 | 8.40 | 0.04500 | 0.37800 |
| Fuel oil | 13.00 | 5.46 | 0.01875 | 0.10238 |
| Loss | 2.00 | 0.84 | | |
| Total | 100.00 | 42.00 | ..... | $1.88566 |
| Glenn Pool Crude:[c] | | | | |
| Gasoline | 17.69 | 7.43 | $0.07081 | $0.52612 |
| Kerosene | 17.28 | 7.26 | 0.04796 | 0.34819 |
| Fuel oil | 60.64 | 25.47 | 0.01875 | 0.47756 |
| Loss | 4.39 | 1.84 | | |
| Total | 100.00 | 42.00 | ..... | $1.35187 |
| Cushing Crude:[c] | | | | |
| Gasoline | 30.90 | 12.98 | $0.07081 | $0.91911 |
| Kerosene | 25.00 | 10.50 | 0.04796 | 0.50358 |
| Gas oil | 15.00 | 6.30 | 0.04500 | 0.28350 |
| Fuel oil | 27.00 | 11.34 | 0.01875 | 0.21263 |
| Loss | 2.10 | 0.88 | | |
| Total | 100.00 | 42.00 | ..... | $1.91882 |
| Average Oklahoma Crude:[c] | | | | |
| Gasoline | 19.52 | 8.20 | $0.07081 | $0.58064 |
| Kerosene | 18.13 | 7.61 | 0.04796 | 0.36498 |
| Gas oil | 2.41 | 1.01 | 0.04500 | 0.04545 |
| Fuel oil | 58.33 | 24.50 | 0.01875 | 0.45938 |
| Loss | 1.61 | 0.68 | | |
| Total | 100.00 | 42.00 | ..... | $1.45045 |

[a] Yields shown by refinery runs at the Fort Worth refinery of the Pierce-Fordyce Oil Association.

[b] Yields shown by refinery runs at the Beaumont refinery of the Magnolia Petroleum Company.

[c] Yields shown by refinery runs at refineries located in Oklahoma and Kansas.

[1] "Conditions in the Healdton Oil Field," Bureau of Corporations, Washington, D. C., 1915.

United States Geological Survey, was the only one, until recently, who had published any extensive results. His samples were collected in the spring of 1908 and therefore do not include the Cushing oils. In 1914, however, the Corporation Commission of Oklahoma decided that, in view of the rapid reduction of the market value of crude oil by the Prairie Oil and Gas Company and other corporations, it should, among other things, attempt to regulate the price of this commodity. In order to carry out their plans intelligently, the Commission asked I. C. Allen, an oil chemist of the United States Bureau of Mines, to analyze samples of oils from the different fields in the State.

The results obtained by Allen were as follows:

TABLE XLIV.—COMPARATIVE ANALYSES OF OKLAHOMA OILS

| | (1) Oklahoma pools. Average of 139, excluding (2), (3) and (4) | (2) Cushing pool. (Bartlesville sand only). Composite of 6 samples | (3) Boston pool. Composite of 5 samples | (4) Healdton pool. Average of 20 samples |
|---|---|---|---|---|
| Specific gravity at 15°C. | 0.8544 | 0.8190 | 0.8330 | 0.8666 |
| Corresponding gravity, Baumé | 33.96 | 46.94 | 38.07 | 31.57 |
| Calories per gram | 10,870.0 | 10,975.0 | 10,927.0 | 10,785.0 |
| B.t.u. per pound | 19,567.0 | 19,755.0 | 19,661.0 | 19,414.0 |
| Viscosity at 20°C. (Engler) | 3.9 | 1.3 | 1.5 | 3.0 |
| Water, per cent | 0.0 | trace | 0.1 | trace |
| Sulphur, per cent | 0.23 | 0.22 | 0.15 | 0.70 |
| Gasoline, per cent. | | | | |
| By weight | 8.1 (59.6°Bé.) | 25.8 (58.5°Bé.)[1] | 18.7 (58.6°Bé.)[1] | 6.0 (57.7°Bé.) |
| By volume, or | 9.4 | ..... | ..... | 7.0 |
| By weight | 13.8 (56.3°Bé.) | 32.9 (55.4°Bé.)[1] | 26.5 (55.4°Bé.)[1] | 11.2 (54.4°Bé.) |
| By volume | | | | |
| Kerosene, per cent. | | | | |
| By weight | 38.5 (42.2°Bé.) | 32.0 (42.2°Bé.)[1] | 32.8 (42.2°Bé.)[1] | 28.8 (42.2°Bé.) |
| By volume, or | 40.5 | ..... | ..... | 30.7 |
| By weight | 40.5 | | | |
| By volume | 24.2 (42.1°Bé.) | 20.4 (41.7°Bé.)[1] | 22.1 (41.7°Bé.)[1] | 20.2 (41.3°Bé.) |
| Lubricants, per cent. | | | | |
| By weight | 26.1 (29.1°Bé.) | 23.2 (29.5°Bé.)[1] | 29.5 (29.5°Bé.)[1] | 29.7 (29.9°Bé.) |
| By volume, or | 25.3 | ..... | ..... | 29.4 |
| By weight | 34.7 (30.0°Bé.) | 27.7 (31.7°Bé.)[1] | 32.4 (31.7°Bé.)[1] | 33.1 (33.4°Bé.) |
| Residue, per cent. | | | | |
| By weight | 25.6 | 17.5 | 16.8 | 34.4 |
| Loss | | | | |
| By weight | 0.8 | 1.5 | 2.2 | 1.1 |
| Total | 100.0 | 100.0 | 100.0 | 100.0 |

[1] Estimated.

H. H. Willock, of Pittsburgh, Pa., made the following report to the Corporation Commission on the values of the Cushing crude as compared to those of the Pennsylvania crude:

"As near as I could figure, the values of the products from Pennsylvania and Cushing crudes, based on the comparative run made by the Wells Refining Oil Process Company, would be as follows:

PENNSYLVANIA OIL

| | °Bé. | | | |
|---|---|---|---|---|
| Gasoline | 66.2, | 25 gal. at | $0.12 | $3.00 |
| Turpentine substitute | 51.9, | 15 gal. at | 0.08½ | 1.28 |
| Kerosene | 45.7, | 15 gal. at | 0.05 | 0.75 |
| 300° oil | 40.3, | 15 gal. at | 0.05 | 0.75 |
| Non-viscous neutral oil | 35.5, | 12 gal. at | 0.04½ | 0.54 |
| Viscous neutral oil | 31.0, | 8 gal. at | 0.12 | 0.96 |
| Steam-refined cylinder stock | 25.0, | 8 gal. at | 0.12 | 0.96 |
| Refined paraffin wax | | 2 gal. at | 0.25 | 0.50 |
| Total | | 100 | | $8.74 |
| 5 per cent. gallonage loss in manufacture | | | | 0.44 |
| Total value of products | | | | $8.30 |

CUSHING OIL

| | | | | |
|---|---|---|---|---|
| Gasoline | 65.7, | 30 gal. at | $0.12 | $3.60 |
| Turpentine substitute | 48.2, | 20 gal. at | 0.08½ | 1.70 |
| Kerosene | 40.1, | 15 gal. at | 0.03 | 0.45 |
| Gas oil | 34.6, | 15 gal. at | 0.02 | 0.30 |
| Viscous neutral oil | 28.0, | 10 gal. at | 0.10 | 1.00 |
| Steam-refined cylinder stock | 24.0, | 6 gal. at | 0.08 | 0.48 |
| Refined paraffin wax | | ½ gal. at | 0.25 | 0.13 |
| Asphalt | | 3½ gal. at | 0.06 | 0.21 |
| Total | | 100 | | $7.87 |
| 5 per cent. gallonage loss in manufacture | | | | 0.39 |
| Total value of products | | | | $7.48 |

"You will note from the above figures that the products from 100 gal. of Pennsylvania oil only exceed in value the products of a like number of gallons of Cushing crude oil by 82 cts., or, in other words, that Pennsylvania oil, from a refining standpoint, is worth only approximately 10 per cent. more than the Cushing oil, although, at the present time, with Cushing oil selling at 75 cts. and Pennsylvania oil at $1.90 at the wells, the price of Pennsylvania oil is over two and one-half times that of Cushing oil."

The following tests made on Cushing crude oil also give an idea of the character of the petroleum.[1]

RESULT OF A TEST RUN ON 30,000 GAL. OF 40.9°BÉ. CUSHING CRUDE, FROM BARTLESVILLE, WHEELER, AND LAYTON SANDS

| | |
|---|---|
| Crude benzine | 36.0 |

80 per cent. of this, if rerun, would be finished 60 per cent. gasoline.

| | |
|---|---|
| Kerosene | 20.0 |
| Gas oil | 10.0 |
| Wax distillate | 21.0 |
| Residuum | 9.0 |

Layton crude of 43.5°Bé.

| | |
|---|---|
| Gasoline, 60° to 61° gravity | 50.0 |
| Water-white, 40° to 41° gravity | 12.5 |
| Residuum or road base | 33.5 |
| Loss | 4.0 |

TEST OF 580 BBL. OF CUSHING CRUDE (40°BÉ.) FROM BARTLESVILLE, WHEELER, AND LAYTON SANDS

207.54 bbl. crude benzine, or 35.78 per cent.
96.66 bbl. water-white distillate, or 16.67 per cent.
177.68 bbl. wax distillate, or 30.64 per cent.
70.37 bbl. residuum, or 13.51 per cent.
19.7 bbl. loss, or 3.4 per cent.

WHEELER CRUDE (41.5°BÉ.)

| | |
|---|---|
| Gasoline, 60° to 61° gravity | 37.5 |
| Water-white, 40° to 41° gravity | 21.0 |
| Wax distillate | 26.0 |
| Tar, or heavy residuum | 12.0 |
| Loss | 5.5 |

Records obtained from the Superintendent of Cosden & Company, Tulsa, Oklahoma.

J. E. Ellis, Superintendent of the Cushing Refining Company at Cushing, in discussing the crude oils of the Cushing fields, stated that the average gravities of the Layton, Bartlesville, and Wheeler crudes received at his refinery are about 41°, 40.5°, and 38° to 39°Bé., respectively. The tests at his plant also show that the Layton crude is slightly better than Bartlesville crude, inasmuch as it yields about 5 per cent. more benzine; but, since this is really the only difference, they have practically the same value. In general, the Wheeler crude proves to be of a

[1] BUTTRAM, *loc. cit.*, 58.

MAGNOLIA PETROLEUM COMPANY'S STATEMENT OF COMPARATIVE REFINING COSTS (FIGURES TAKEN FROM CORSICANA REFINERY MONTHLY STATEMENTS)

March, 1914

| | | |
|---|---|---|
| Electra crude run, gallons | 2,225,201 | |
| Corsicana light crude run, gallons | 258,203 | |
| Total crude run | 2,483,404 | |
| Refined oil and naphtha cut from total crude run: | | |
| Refined oil, gallons | 760,966 : 30.64 per cent. | |
| Naphtha, gallons | 640,598 : 25.80 per cent. | |
| Cost of distillation, $7,907.37 | | |
| Cost of distilling 1 gal. | $0.005641 | |
| Cost of treating 1 gal. | 0.001885 | |
| Cost of steam-stilling 1 gal. | 0.000404 | |
| Total manufacturing cost 1 gal. refined oil and naphtha | $0.007930 | $0.007930 |

April, 1914

| | | |
|---|---|---|
| Electra crude run, gallons | 90,707 | |
| Healdton crude run, gallons | 4,097,904 | |
| Corsicana light crude run, gallons | 74,198 | |
| Total crude run | 4,262,809 | |
| Refined oil and naphtha cut from total crude run: | | |
| Refined oil, gallons | 917,190 : 21.52 per cent. | |
| Naphtha, gallons | 565,278 : 13.26 per cent. | |
| Total cuts | 1,482,468 | |
| Cost of distillation, $9,318.59. | | |
| Cost of distilling 1 gal. | $0.006285 | |
| Cost of treating 1 gal. | 0.001865 | |
| Cost of steam-stilling 1 gal. | 0.000480 | |
| Total manufacturing cost, 1 gal. refined oil and naphtha | $0.008630 | |
| The 917,190 gal. refined oil shown above were pumped to Beaumont and rerun and treated again at an estimated cost of $4,585.95. This amount figured against the total cuts shown above, 1,482,468 gal. | 0.003093 | |
| Or a total manufacturing cost per gallon of product of | $0.011723 | $0.011723 |
| Difference in cost, March and April | | $0.003793 |

This difference was said to be equal to the cost of removing the sulphur from 1 gal. of refined product from Healdton crude.

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slightly lower grade of oil than the Layton and Bartlesville crudes. Again, the Layton and the Bartlesville oils are usually of a light green color, while the Wheeler usually has a darker shade of green than that of the other two oils.

TABLE XLV.—STATEMENT OF THE MAGNOLIA PETROLEUM COMPANY, SHOWING AN EXPERIMENT WITH 1 BBL. OF HEALDTON CRUDE FOR THE PURPOSE OF DETERMINING IF ENGINE OILS CAN BE MANUFACTURED THEREFROM

1 Bbl. Healdton Crude for Corsicana, Gravity 30.6°Bé.

| Cuts | Per cent. | Temperature | | Gravity °Bé. | Chill | |
|---|---|---|---|---|---|---|
| | | Still | Vapor | | | |
| 1 | 2.5 | 360 | 260 | 61.0 | ..... | Naphtha, 7½ per cent. |
| 2 | 2.5 | 390 | 290 | 58.1 | ..... | Naphtha, 7½ per cent. |
| 3 | 2.5 | 420 | 330 | 55.2 | ..... | Naphtha, 7½ per cent. |
| 4 | 2.5 | 450 | 360 | 51.6 | ..... | Light end, common oil, 2½ per cent. |
| 5 | 2.5 | 470 | 380 | 48.7 | ..... | Water-white stock, 15 per cent. |
| 6 | 2.5 | 500 | 400 | 46.8 | ..... | Water-white stock, 15 per cent. |
| 7 | 2.5 | 520 | 420 | 45.1 | ..... | Water-white stock, 15 per cent. |
| 8 | 2.5 | 540 | 440 | 43.3 | ..... | Water-white stock, 15 per cent. |
| 9 | 2.5 | 560 | 460 | 41.8 | ..... | Water-white stock, 15 per cent. |
| 10 | 2.5 | 590 | 490 | 40.5 | ..... | Water-white stock, 15 per cent. |
| 11 | 2.5 | 600 | 510 | 39.5 | ..... | Heavy end, common oil, 7½ per cent. |
| 12 | 2.5 | 619 | 520 | 39.0 | ..... | Heavy end, common oil, 7½ per cent. |
| 13 | 2.5 | 634 | 534 | 37.9 | ..... | Heavy end, common oil, 7½ per cent. |
| 14 | 2.5 | 650 | 559 | 37.5 | ..... | Gas oil distillate, 27.5 per cent. |
| 15 | 2.5 | = | 562 | 36.5 | ..... | Gas oil distillate, 27.5 per cent. |
| 16 | 2.5 | = | 563 | 36.5 | 16 | Gas oil distillate, 27.5 per cent. |
| 17 | 2.5 | = | 565 | 36.7 | 18 | Gas oil distillate, 27.5 per cent. |
| 18 | 2.5 | = | 570 | 36.8 | 22 | Gas oil distillate, 27.5 per cent. |
| 19 | 2.5 | = | 571 | 36.7 | 18 | Gas oil distillate, 27.5 per cent. |
| 20 | 2.5 | = | 572 | 35.6 | 26 | Gas oil distillate, 27.5 per cent. |
| 21 | 2.5 | = | 573 | 34.4 | 26 | Gas oil distillate, 27.5 per cent. |
| 22 | 2.5 | = | 589 | 34.1 | 26 | Gas oil distillate, 27.5 per cent. |
| 23 | 2.5 | = | 595 | 34.5 | 26 | Gas oil distillate, 27.5 per cent. |
| 24 | 2.5 | = | 591 | 35.2 | 26 | Gas oil distillate, 27.5 per cent. |
| 25 | 2.5 | = | 591 | 35.2 | 28 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 26 | 2.5 | = | 591 | 36.5 | 32 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 27 | 2.5 | = | 591 | 38.5 | 40 | 30 per cent. paraffin distillate; gravity 33.1°; pour, 40. |
| 28 | 2.5 | = | 591 | 37.5 | 32 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 29 | 2.5 | = | 592 | 36.5 | 70 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 30 | 2.5 | = | 593 | 37.0 | 42 | 30 per cent. paraffin distillate; gravity 33.1°; pour, 40. |
| 31 | 2.5 | = | 593 | 37.5 | 70 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 32 | 2.5 | = | 597 | 35.3 | 82 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 33 | 2.5 | = | 600 | 32.5 | 76 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 34 | 2.5 | = | 600 | 32.2 | 84 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 35 | 2.5 | = | 600 | 32.1 | 86 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40. |
| 36 | 2.5 | = | 600 | 30.9 | 90 | 30 per cent. paraffin distillate; gravity, 33.1°; pour, 40; |
| 37 | 2.5 | = | 600 | 18.0 | 112 | Wax tailings, 2½ per cent. |
| Bottom | ..... | ..... | ..... | ..... | ..... | Coke, 4.72 per cent., 13¾ lb. Loss, 2.78 per cent. |

The paraffin distillate was run in a 1-bbl. still; overhead, 41.07 per cent.; gravity, 40°. Bottom, 58.93 per cent. heavy oil and paraffin; gravity 27.0°; flash, 325°; viscosity, 77; pour, 60.

Bottom pressed over at 40°F. = 92.74 per cent. lubricating distillate; gravity, 27.0°; flash, 325°; viscosity, 77; pour, 35.

Slack wax, 7.26 per cent.; m.p., 112°F.

Slack wax, treated with 10 lb. acid: sweated to m.p. produced 63 per cent. of foots oil and 37 per cent. of 128° m.p. wax.

The 92.74 per cent. lubricating distillate gave the following:

| Cut | Per cent. | Temperature | Gravity °Bé. | Flash °F. | Viscosity | Chill | |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 600 | 31.5 | 270 | 42 | 46 | |
| 2 | 5 | 605 | 30.4 | 275 | 45 | 46 | All overhead cuts mixed for pale oil: |
| 3 | 5 | 610 | 29.6 | 275 | 49 | 48 | |
| 4 | 5 | 615 | 28.1 | 280 | 54 | 46 | Gravity, 28.2° Bé.; flash, 310°F.; viscosity, 72; chill, 46°F. |
| 5 | 5 | 620 | 27.9 | 285 | 56 | 44 | |
| 6 | 5 | 622 | 27.5 | 290 | 57 | 46 | |
| 7 | 5 | 625 | 27.4 | 300 | 59 | 48 | Treated with 10 lb. 66° sulphuric acid: |
| 8 | 5 | 628 | 27.3 | 305 | 60 | 46 | gravity, 28.2° Bé.; flash, 310°F.; viscosity, 60; chill, 50°F.; color, 890. |
| 9 | 5 | 630 | 27.2 | 310 | 61 | 46 | |
| 10 | 5 | 635 | 26.9 | 315 | 62 | 46 | |
| 11 | 5 | 630 | 26.8 | 320 | 66 | 48 | |
| 12 | 5 | 630 | 26.2 | 330 | 74 | 46 | |
| 13 | 5 | 630 | 26.1 | 335 | 82 | 44 | |
| 14 | 5 | 635 | 25.8 | 340 | 96 | 46 | |
| 15 | 5 | 640 | 25.4 | 345 | 96 | 46 | |

Bottom, 25 per cent.; gravity, 22.3° Bé.; flash, 390°F.; viscosity, 189; pour, 35. (Color, A Re(d)).

Treated with 15 lb. of 66° acid = gravity, 22.4° Bé.; flash, 390.0°F.; viscosity, 142; chill, 46°F.

Paraffin distillate worked up = 12.30 per cent. of overhead of h.o. and p. 12.30 per cent. pale oil, 4.10 per cent. red oil, 0.82 per cent. foots oil, 0.48 per cent. finished wax.

Note.—Yield of Healdton crude oil, shown in percentages of the volume of the crude refined:

| | Per cent. |
|---|---|
| Naphtha | 7.50 |
| W. W. 150° | 15.00 |
| Common oil | 10.00 |
| Gas oil | 12.32 |
| Foots oil | 0.81 |
| Pale oil | 12.30 |
| Red oil | 4.10 |
| Finished wax | 0.47 |
| Fuel oil | 27.50 |
| Wax tailings | 2.50 |
| Coke | 4.72 |
| Loss | 2.78 |
| | 100.00 |

TABLE XLVI.—MID-CONTINENT REFINERIES AND THEIR INVESTMENT (1915)

KANSAS

| Name of company and year built | Total investment |
|---|---|
| Miller Petroleum Refining Co., Humboldt, 1906 | $73,626 |
| Kansas City Refining Co., Kansas City, 1906 | 250,000 |
| Eastern Kansas Oil Co., Moran, 1905 | 300,000 |
| Chanute Refining Co., Chanute, 1907 | 300,000 |
| Kansas Co-operative Refining Co., Chanute, 1906 | 200,000 |

TABLE XLVI.—MID-CONTINENT REFINERIES AND THEIR INVESTMENT (1915).—*Continued*

KANSAS

| Name of company and year built | Total investment |
|---|---|
| Great Western Oil Refining Co., Erie, 1905 | 754,000 |
| Kansas Oil Refining Co., Coffeyville, 1906 | 300,000 |
| Niotaze Refining Co., Niotaze, 1905 | 275,000 |
| Cudahy Refining Co., Coffeyville, 1908 | 1,400,000 |
| National Refining Co., Coffeyville, 1907 | 500,000 |
| Uncle Sam Oil Co., Cherryvale, 1906 | 125,000 |
| Petroleum Products Co., Independence, 1910 | 2,500,000 |
| Kanotex Refining Co., Caney, 1906 | 650,000 |
| Kansas Crude Oil Co., Chanute, 1909 | 12,000 |
| Lesh Refining Co., Arkansas City, 1914 | 30,000 |
| Rollin Oil Refinery, Rollin, 1908 | 20,000 |
| Wichita Refining Co., Wichita, 1914 | 10,000 |
| Hutchinson Refining Co., Hutchinson, 1915 | 5,000 |
| Rosedale Refining Co., Rosedale, 1915 | 25,000 |

OKLAHOMA

| | |
|---|---|
| Cudahy Refining Co., Muskogee, 1905 | $95,000 |
| Muskogee Refining Co., Muskogee, 1906 | 350,000 |
| American Refining Co., Okmulgee, 1906 | 583,100 |
| Oklahoma Refining Co., Oklahoma City, 1906 | 280,667 |
| Uncle Sam Oil Co., Tulsa, 1906 | 50,000 |
| Sapulpa Refining Co., Sapulpa, 1908 | 749,907 |
| Indiahoma Refining Co., Okmulgee, 1910 | 1,258,000 |
| Milliken Refining Co., Vinita, 1910 | 1,300,000 |
| Constantin Refining Co., Tulsa, 1911 | 400,000 |
| Webster Refining Co., Coalton, 1911 | 15,000 |
| Consumers' Refining Co., Cushing, 1912 | 464,791 |
| Consumers' Refining Co., tank cars | 300,000 |
| Consumers' Refining Co., pipe lines | 100,000 |
| Ponca Refining Co., Ponca City, 1912 | 600,000 |
| Cushing Refining Co., Cushing, 1913 | 445,000 |
| Cosden & Co., Tulsa, 1912 | 1,500,000 |
| Cosden & Co., Cushing, 1911 | 100,000 |
| Cosden & Co., Bigheart, 1908 | 80,000 |
| Consolidated Oil Refining Co., Cleveland, 1913 | 100,000 |
| Phœnix Refining Co., Sand Springs, 1913 | 300,000 |
| Pierce Oil Corporation, Sand Springs, 1913 | 600,000 |
| Texas Company, Tulsa, 1913 | 1,200,000 |
| Chanute Refining Co., Cushing, 1914 | 600,000 |
| Ardmore Refining Co., Ardmore, 1914 | 100,000 |
| Illinois Oil Refining Co., Cushing, 1914 | 65,000 |
| Peerless Oil Refining Co., Cushing, 1914 | 600,000 |
| Hillman Refining Co., Cushing, 1914 | 12,000 |
| Continental Refining Co., Bristow, 1914 | 20,000 |
| Webster Oil and Gasoline Co., Yale, 1915 | 17,030 |

TABLE XLVI.—MID-CONTINENT REFINERIES AND THEIR INVESTMENT (1915).—*Continued*

OKLAHOMA

| Name of company and year built | Total investment |
|---|---|
| International Refining Co., Cushing, 1915 | 200,000 |
| Sun Oil Co., Yale, 1915 | 100,000 |
| North American Refining Co., Pemeta, 1915 | 150,000 |
| Capital Refining Co., Oklahoma City, 1915 | 20,000 |
| Crystal White Refining Co., Allen, 1915 | 10,000 |
| Southern Oil Corporation, Yale, 1915 | 60,000 |
| Portland Oil and Refining Co., Okmulgee, 1915 | 10,000 |
| OTHER STATES | |
| Indiahoma Refining Co., East St. Louis, Ill., 1907 | $100,000 |
| Consolidated Oil Refining Co., East St. Louis, Ill., 1909 | 50,000 |
| Consolidated Oil Refining Co., East St. Louis, Ill., 1915 | 35,000 |
| Wilhoit Refining Co., Joplin, Mo., 1913 | 120,000 |
| St. Joseph Refining Co., St. Joseph, Mo., 1915 | 15,000 |
| Magnolia Petroleum Co., Fort Worth, Tex., 1914 | 457,840 |
| Producers' Refining Co., Gainesville, Tex., 1915 | 683,000 |
| Total investment | $22,006,761 |

TABLE XLVII.—STANDARD OIL COMPANY PLANTS USING MID-CONTINENT CRUDE

| | Barrels daily |
|---|---|
| Standard Oil Co., Neodesha, Kan | 7,500 |
| Standard Oil Co., Sugar Creek, Mo | 12,500 |
| Standard Oil Co., Baton Rouge, La | 12,000 |
| Standard Oil Co., Wood River, Ill | 12,500 |
| Standard Oil Co., Whiting, Ind | 30,000 |

**The Products of California Petroleum.**—The petroleum products manufactured in the California field are given in the accompanying list of the refineries of that State.

The customary trade names of the products of California petroleum, are thus defined by the General Petroleum Company:

*Gasoline.*—This is usually of 60°Bé. gravity.

*No.* 1 *Tops.*—This includes gasoline stock and is of 55°Bé. gravity.

*No.* 1 *Engine Distillate,* which possesses a gravity of 50°Bé.

*No.* 2 *Engine Distillate,* which is water-white to light amber in color and has a gravity of 45°Bé.

*Kerosene Stock,* amber in color and of 40° to 42°Bé. gravity.

*Stove Distillate,* dark in color and of 30° to 34°Bé. gravity.

*Orchard Heating Oil* (*Smudge Oil*).—A dark product of 26°

to 28°Bé. gravity, used in the orange and lemon groves to prevent frost from damaging the trees.

*Fuel Oil.*—This product usually has a gravity of 17°Bé. and a flash-point of 150°F., and contains about 30 per cent. of asphalt.

The following information regarding certain commercial uses of California fuel oil, is of interest in this connection.[1]

Fuel Oil on Railroads.—Doubtless the largest consumption of California fuel oil is by the railroads. In 1913, the Southern Pacific alone burned about 32,000 bbl. daily, according to evidence presented to the California Railroad Commission, and the expectation is that each year will see this figure increased by 2,000 to 2,500 bbl.

Heating value tests on 745 locomotives show 1 ton (2,000 lb.) of ordinary bituminous coal (13,350 B.t.u.) equal to 3.62 bbl. of oil, and on eleven steamboats running six months 3.49 bbl.[2]

Consumption in running over the Sierra Nevada Mountains was from 35 to 49 gal. per 1,000-ton miles. Usually the oil is 14° to 15°Bé., but the specifications are very broad, covering the five following points: (1) Viscosity, such as to flow freely through a 4-in. pipe at 70°F.; (2) water content, less than 2 per cent., using gasoline in the testing machine; (3) flash, less than 110° Tagliabue; (4) temperature standard, 60°F., with a correction of 1 per cent. for every 25°F.; (5) gravity, 14° to 29°Bé.

Oil for Gas Generation.—On the list of large consumers of crude oil the gas manufacturers stand near the head. There were 56 gas plants using oil as early as 1910.[3] The rate of consumption per thousand feet of gas generated is 8.13 to 8.55 gal., about 15 per cent. to 16 per cent. of that amount being consumed as fuel for the retorts. The most suitable oil is said to be of the gravity 14° to 17°Bé., with less than 1 per cent. sulphur.

Panama Canal.—The Panama Canal construction consumed considerable oil, a six-year contract having been made in 1909 with the Union Oil Company at $1.10, the monthly deliveries being 30,000 to 60,000 bbl. At such a price the oil was said to be cheaper than Pocahontas coal at $6.24.

Displacement of Coal.—The amount of coal imported and used in California has declined greatly since the year 1900, when the

[1] *Bull.* **69**, *Cal. State Min. Bur.*, **1914**, 83. See also *Report No.* **291** (**1**, 285 *et seq.*) *of the Mines Branch, Canada Dept. of Mines.*

[2] STILLMAN, *Trans. Am. Soc. Mech. Eng.*, **33** (1911), 887.

[3] JONES, *J. Elec., Power and Gas*, **24**, 492.

Kern River oil field was developed. The importation of coal in the year 1865 was 150,141 tons, and the amount increased steadily to 1,889,128 tons in 1900, when a rapid decline began and imports are now less than 500,000 tons. In other words, oil has actually displaced at least 1,400,000 tons of coal, and at the rate at which the importation steadily increased from 1865 to 1900 (50,000 tons per year) the imports would now probably have been 2,550,000 tons; hence the displacement is probably as high as 2,000,000 tons per year, or some seven million barrels of oil are being burned annually in California in place of coal. However, recent reports state that the proportion of petroleum burned decreased from about 50 per cent. in 1912 to about 25 per cent. in 1914.

Diesel Engines.—The development of Diesel engines doubtless opens new fields for oil consumption and bids fair to become a very important factor. The engine operation will not be described further than to say that fuel oil is sprayed into the cylinder and ignites, due to the heat of the compressed air. The oil does not explode, as does gasoline in an engine, but simply ignites and the combustion products expand.

Fuel used for these engines, when dependent upon California products, is usually a treated oil turned out by the refineries, and selling under various trade names as fuel oil.

One of the usual oils gave the following analysis:

24°Bé. (specific gravity, 0.9091).
Open flash, 180°F.
Closed flash, 165°F.
Burning point, 235°F.
Asphaltum, 25 per cent.
Sulphur, 0.75 per cent.
British thermal units per pound, 19,200.

The consumption of fuel varies from 0.525 lb. to 0.721 lb. per brake horsepower-hour.

The United States Bureau of Mines has dealt more fully with the subject of heavy fuel oil for internal combustion engines. *Technical Paper* **37** of that Bureau shows that 1 lb. of oil used in a Diesel equals 2½ lb. of oil or 4 lb. of coal, used in generating steam for a turbine, to exert the same power.[1]

[1] On the products of combustion of fuel oils, see *Rept. Chicago Assn. of Commerce Committee of Inves. on Smoke Abatement and Electrification of Railway Terminals*, **1915**, pp. 191–193. The subject of liquid fuels will be considered at length by the authors in their treatise on "American Fuels" (McGraw-Hill Book Co., **1918**).

TABLE XLVIII.—CALIFORNIA OIL REFINERIES AND TOPPING PLANTS[1]

(Operating or Capable of Operation in 1914)

| Name | Location | Date built | Capacity per month. Crude, barrels. Asphalt, tons | Stills | | Products |
|---|---|---|---|---|---|---|
| | | | | Number | Size (barrels) | |
| Adeline Oil Company | Maricopa | 1910 | ..... | 2 | 150 | Distillate, road oil. |
| American Oriental Oil Company | Martines | 1904 | 1,000 tons | ..... | ..... | Distillate, asphalt. |
| American Petroleum Company | Fellows | 1913 | ..... | Continuous system | | |
| Amalgamated Oil Company | Los Angeles | 1905 | 105,000 bbls. | 3 | 300 | Distillate, road oil. |
| Asphaltum Oil and Refining Company | Los Angeles | ..... | 14,000 bbl. | 1<br>2<br>2 | 200<br>150<br>50 | Kerosene, distillate, stove oil, gasoline, asphalt. |
| Associated Oil Company | Gaviota | 1899 | 150,000 bbl. | 9<br>3 | 220<br>200 | Fuel oil, gasoline, kerosene, and distillate. |
| Associated Oil Company | Avon | 1913 | 360,000 bbl. | 8 | 500 | Fuel oil, gasoline, distillate. |
| Atlas Refinery | Los Angeles[2] | 1900 | 30,000 bbl.<br>1,400 tons | 2<br>1 | 300<br>265 | Distillate, road oil. |
| Barber Asphalt Paving Company | Los Angeles | 1890 | 30,000 bbl.<br>1,400 tons | 3<br>3 | 300<br>150 | Distillate, asphalt, stove oil. |
| Beckett | Arroyo Grande | | | | | |
| Buckeye Refining Company | Bakersfield[3] | 1901 | ..... | ..... | ..... | Light oils. |
| California Fresno Oil Company | Fresno | 1882 | 100 tons | 4 | 75 | Asphalt. |
| California Liquid Asphalt Company | Hadley | 1909 | 1,000 tons<br>13,000 bbl. | 2<br>1 | 100<br>250 | Distillate, kerosene, gasoline, and asphalt. |

[1] On the refining of California petroleum, see STABLER, *Cal. Derrick*, 6 (1914), Nos. 1 and 2; HOWARD, *Western Eng.*, 2 (1913), 223; ROBINSON, *ibid.*, 3 (1913), 47; and *Bull. No.* 69, *Cal. State Min. Bur.*, 1914. On methods for removing light oils from crude oils, see *Cal. Oil World*, 3, No. 42, 11; and BELL, *op. cit.*

[2] Leased to Union Oil Company.

[3] Leased to National Oil Refining and Manufacturing Company.

TABLE XLVIII.—CALIFORNIA OIL REFINERIES AND TOPPING PLANTS.—*Continued*

| Name | Location | Date built | Capacity per month. Crude, barrels. Asphalt, tons | Stills | | Products |
|---|---|---|---|---|---|---|
| | | | | Number | Size (barrels) | |
| California Oil and Asphalt Company | Los Angeles | 1902 | 450 tons | 2 | 100 | Distillate, asphalt. |
| Capitol Refining Company | Berkeley | 1900 | 600 tons | ..... | ..... | Distillate, asphalt. |
| Capitol Crude Oil Company | Santa Paula | | | | | |
| Columbia Oil, Asphalt and Refining Company | Carpinteria | 1905 | 450 tons | 2<br>1 | 150<br>250 | Asphalt, distillate. |
| Continental Oil Company | Los Angeles | 1907 | 600 tons | ..... | ..... | Asphalt, distillate. |
| Cooper | San Diego | | | | | |
| Densmore-Stabler Refining Company | Los Angeles[1] | 1902 | 20,000 bbl. | 6 | 150 | Distillate, gasoline, stove oil, asphalt, kerosene, lubricants. |
| Eastern Consolidated Oil Company | Bakersfield | 1904 | 1,000 tons | ..... | ..... | Distillate, asphalt. |
| El Oso Asphalt Company | ..... | ..... | 9,000 bbl. | 1<br>1<br>1 | 235<br>160<br>30 | Distillate, asphalt, gasoline. |
| Ensign-Baker Refining Company | Hadley | 1910 | 1,000 tons | ..... | ..... | Distillate, asphalt. |
| General Petroleum Company | Vernon | 1913 | 300,000 bbl. | Trumble plant | | Gasoline, distillate, fuel oil. |
| General Petroleum Company | Mojave | 1913 | 200,000 bbl. | Trumble plant | | Gasoline, distillate, fuel oil. |
| General Petroleum Company | Kerto | 1913 | 2,000 bbl. | Trumble plant | | Gasoline, distillate. |
| Guaranty Oil Company | Los Angeles | ..... | 30,000 bbl. | 5 | 200 | Gasoline, distillate. |
| Hercules Oil Refining Company[2] | Los Angeles | 1900 | 30,000 bbl. | 4<br>2<br>2 | 100<br>300<br>600 | Distillate, asphalt, road oil, stove oil. |

[1] Run by Turner Oil Company.
[2] Fairchild, Gilmore, Wilton Company.

TABLE XLVIII.—CALIFORNIA OIL REFINERIES AND TOPPING PLANTS.—*Continued*

| Name | Location | Date built | Capacity per month. Crude, barrels. Asphalt, tons | Stills | | Products |
|---|---|---|---|---|---|---|
| | | | | Number | Size (barrels) | |
| Jordan Oil Company | Los Angeles | 1900 | 9,000 bbl. | 2<br>1 | 125<br>250 | Distillate, kerosene, lubricants. |
| King Refining Company | Bakersfield | 1901 | 500 tons | ..... | ..... | Distillate, asphalt. |
| Los Angeles Oil Refining Company | Olinda | ..... | ..... | Continuous system | | |
| Monarch Oil Refining Company | Berkeley | 1910 | 600 tons | ..... | ..... | Distillate, asphalt. |
| More | Goleta | | | | | |
| National Oil Refining and Manufacturing Company | Bakersfield | 1904 | 1,200 tons | ..... | ..... | Distillate, asphalt. |
| Pacific Refining and Roofing Company | San Francisco | 1903 | 300 tons | ..... | ..... | Roofing, asphalt. |
| Pacific States Refining Company | Fruitvale | 1904 | 400 tons | ..... | ..... | Distillate, asphalt. |
| Paraffin Paint Company | South San Francisco | 1900 | 100 tons | 2<br>2 | 125<br>30 | Paint and roofing asphalt. |
| Phoenix Refining and Manufacturing Company | Bakersfield | 1902 | 1,200 tons | ..... | ..... | Distillate, asphalt. |
| Pinal-Dome Oil Company | Betteravia | 1910 | 36,000 bbl. | ..... | ..... | Fuel oil, distillate, gasoline. |
| Pioneer Roll Paper Company | Los Angeles | 1908 | 14,000 bbl.<br>600 tons | 2 | 300 | Roofing paper asphalt, distillate. |
| Producers Refining Company | Bakersfield | 1903 | 700 tons | | | |
| Producers and Refiners Oil Company | Oilport | 1906 | 5,000 bbl. | 15 | ..... | Never operated. |
| Puente Oil Company | Chino | 1896 | 30,000 bbl. | 3<br>1<br>1<br>1 | 220<br>200<br>150<br>40 | Distillate, fuel oil. |

TABLE XLVIII.—CALIFORNIA OIL REFINERIES AND TOPPING PLANTS.—*Continued*

| Name | Location | Date built | Capacity per month. Crude, barrels. Asphalt, tons | Stills | | Products |
|---|---|---|---|---|---|---|
| | | | | Number | Size (barrels) | |
| Southern Refining Company | Los Angeles | 1900 | 21,000 bbl. | 2<br>2<br>1 | 250<br>100<br>50 | Distillates, gasoline, asphalt, kerosene, stove oil, axle grease. |
| Standard Oil Company | Richmond | 1902 | 1,950,000 bbl. | 100<br>15<br>6 | 1,000 crude<br>250 asphalt<br>1,350 rerun | All classes of products. |
| Standard Oil Company | El Segundo | 1911 | 450,000 bbl.<br>2,250 tons | 20<br>3<br>3 | 1,000 crude<br>600 asphalt<br>1,350 rerun | Asphalt, gasoline, benzine, kerosene, fuel oil. |
| Standard Oil Company | Bakersfield | 1912 | 300,000 bbl. | 3<br>3 | 1,000 crude<br>600 road oil | Distillate, fuel oil. |
| Sunset Oil and Refining Company. | Ostend | 1903 | 60,000 bbl. | 16 | 150 | Distillate, asphalt. |
| Sunset Monarch Oil Company | Maricopa | 1907 | 1,500 tons | ..... | ..... | Distillate and asphalt. |
| Union Oil Company | Oleum | 1895 | 300,000 bbl.<br>4,500 tons | 56 | Continuous system | All classes. |
| Union Oil Company | Bakersfield | 1903 | 3,000 tons | 12 | Continuous system | Distillates, kerosene, fuel oil, and asphalt. |
| Union Oil Company | Fullerton | 1911 | 210,000 bbl. | 12 | Continuous system | Gasoline, distillate, and fuel oil. |
| Union Oil Company | Santa Paula | 1912 | 20,000 bbl. | 8 | Dyer system | Gasoline, distillate, and fuel oil. |
| Union Oil Company | Avila | 1910 | 360,000 bbl. | 12 | Dyer system | Gasoline, distillate, and fuel oil. |
| Volcan Oil Company | Bakersfield | 1901 | 400 tons | ..... | ..... | Distillate and asphalt. |
| Warren Bros. | Rodeo[1] | 1903 | 800 tons | ..... | ..... | Distillate and asphalt. |
| Warren Bros. | Bakersfield[2] | 1904 | 1,500 tons | ..... | ..... | Distillate and asphalt. |
| Yosemite Oil Refining Company | Los Angeles | 1902 | 450 tons | 2 | 100 | Distillate and asphalt. |

[1] Leased from the Western Oil Refining Company.
[2] Leased from the California Kern Oil Company.

## THE DISTRIBUTION OF PETROLEUM PRODUCTS TO THE TRADE

Refined oils are distributed to the trade in bulk in 1,500-gal. tank car compartments, and in 3,600-, 4,500-, 5,500-, 6,000-, 6,500-, 8,000-, 10,000- and 12,500-gal. tank cars; these tank cars are usually loaded so that they will be shell-full at a temperature of 60°F., on which basis all bulk products are generally sold.[1]

The ordinary containers are, of course, wooden or steel barrels; and there are, in addition, the following small receptacles or packages: half barrels, kegs (14 to 17 gal.), 5- and 10-gal. wood-jacket cans, 1-gal. cans (usually 12 in a case), 100-lb. kegs, and 5-, 10-, 25- and 50-lb. slip-top and friction-top (for thin greases) tins. Brief reference will now be made to certain technical factors involved in the marketing of the liquid products[2] of petroleum in receptacles of this group.

**Wooden Barrels.**—The preparation of barrels is an important item in the marketing of refined distillates. Oil barrels must be perfectly tight and free from foreign matter which may contaminate the product shipped therein, and it is therefore necessary that they are properly driven, cleaned, glued and dried. The drying subsequent to gluing is probably the most difficult to accomplish satisfactorily, for the glue is likely to break if dried too rapidly[3] and to mildew if dried too slowly.

In drying with hot air, a fan forces a current of air into an iron chamber inclosing a series of steam pipes, wherein it is heated to the desired temperature, and then forces it through a 10-in. pipe provided with nozzles 30 in. apart. These nozzles are inserted into the glued barrels, and the dry air carries off the water, leaving a coating of glue inside the barrel.

Pickwell[4] has described the barrel-cleaning, gluing and drying arrangements employed at an English petroleum storage installation (see Figs. 173 and 174). At the end of the boiler house is a large air-heating chamber (Fig. 173) containing a coil of steam pipes. The chamber communicates with a blower by a pipe, $AA$, and with the main air-pipe by pipes, $B$. From the main air pipe, three branches lead to three pipes, from which are taken short branches, $E$, of inch pipe, arranged about 2 ft.

[1] See Figs. 159 and 160.

[2] On the distribution of paraffin wax to the trade, see p. 478.

[3] For one elastic composition for use instead of glue, see ANDREWS, English Patent 12827, Sept. 5, 1888.

[4] *Proc. Inst. Civ. Eng.*, **104** (1891), 254.

apart, and so placed as to enter the bungholes of the barrels, which are placed over them in three nests of eight barrels each. Valves,

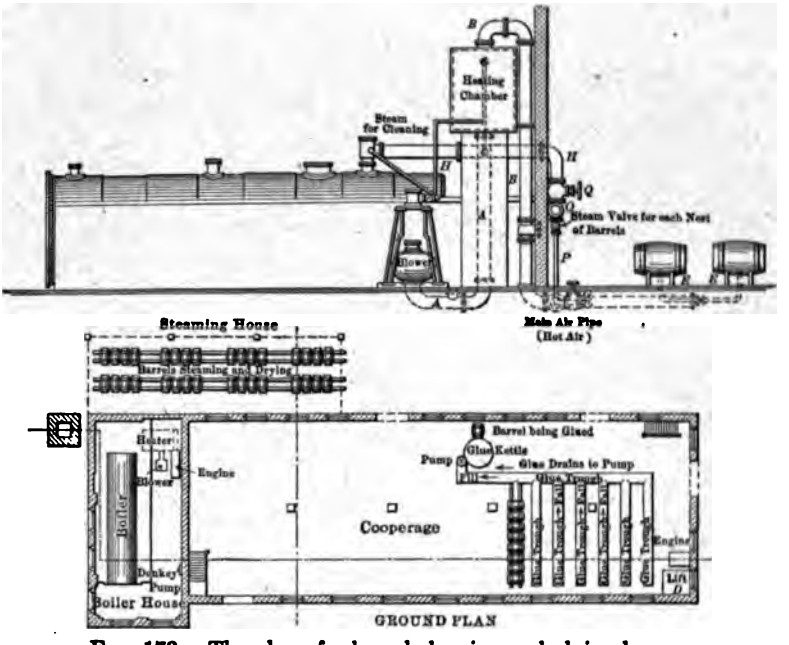



FIG. 173.—The plan of a barrel-cleaning and gluing house.

*G*, permit of shutting off connection with any or all of the nests of barrels. Similarly, steam from the boiler passes through a

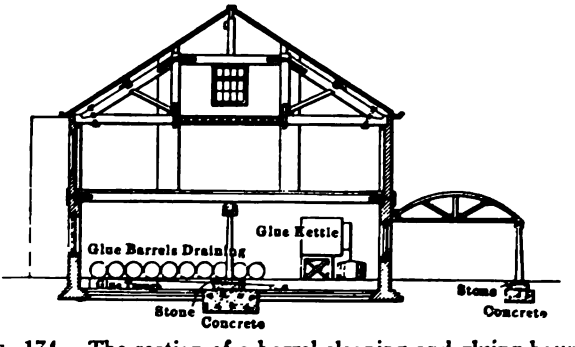



FIG. 174.—The section of a barrel-cleaning and gluing house.

pipe, *H*, into a main, *O*, from which it passes into branches, *P*, leading into the branches of the main air pipe. The barrels are

first steamed for 10 or 15 min., after which the steam is shut off at *Q*; the hot-air valve, *F*, and the branch valves are then opened, and the barrels are dried with hot air. The arrangements for coating the barrels with glue are shown in Fig. 173. After draining, the barrels are lifted to the floor above by a lift, *D*, to be painted. The filling tank in connection with the barrelling house is placed at an altitude of 8 or 10 ft., and the oil flows from it into the barrels through a pipe arranged about 30 in. above the ground, and having branches 7 ft. apart fitted with flexible hose and automatic cocks, so that a number of barrels can be filled simultaneously. As soon as the barrel is sufficiently charged—space being left for expansion of the oil—the supply is automatically cut off; the barrel is then bunged and replaced by an empty one.

**Steel Barrels.**—The manufacture of steel barrels has become an important industry in the last few years, due principally to the increasing demand among the marketers of petroleum oils. A very few years ago the only steel barrels or drums used in this country were those coming from abroad, containing chemicals, but now there are in this country over twenty large concerns manufacturing steel barrels in quantities.[1] Some of these plants are modern in every detail and have a capacity up to 1,000 steel bbl. per day, ranging in sizes from 15 to 110 gal. each. Chemical manufacturers, paint and varnish makers use a great many of these barrels, but it is estimated that 75 per cent. of the output is taken by petroleum refiners.

Steel barrels must stand the continual abuse to which they are subjected. Besides, they must be light in weight, retain their shape and be absolutely free from leakage. Corrosion has now been reduced to a minimum by the method of coating with zinc after the barrel has been made up and by placing the openings in the head of the barrel above the chime; thus water collected on the head does run inside when the barrel stands on end without

[1] Several methods are employed in the manufacture of steel barrels. According to one process, the barrel is formed in two sections, which are joined by pressure, the flanged edges being so formed as to interlock. By another, the bodies are made from rectangular sheets, with a single longitudinal joint welded electrically. Then, too, barrels are stamped cold out of sheet steel and joined circumferentially by spinning the edges of the two shells together.

plug or with loose plug. Barrels so constructed are guaranteed to last many years.[1]

The Interstate Commerce Commission's Bureau for the Safe Transportation of Explosives and Dangerous Articles under the direction of Colonel Dunn has encouraged the use of steel barrels and requires that steel barrels or drums for the shipment of gasoline or other inflammables must stand certain pressure and drop tests and must be made of a specified weight of steel according to the size. Every container used for this purpose must be stamped as complying with specification No. 5. This has brought about a great improvement in lessening the number of accidents and fire losses.

Some of the largest oil companies are rapidly replacing their wooden cooperage with steel which will last many years, and, furthermore, they are relieved of the expense and trouble of recoopering wooden barrels. Besides, the saving in leakage is a considerable item. From a marketing viewpoint there are the following advantages: the customer prefers a steel to a wood barrel on account of possible leakage and fire risk in the case of the latter, and some companies quote him a lower price for oil in steel barrels.

There are many different methods of handling the steel barrel in the service of oil companies: some loan the barrel without charge; others make a charge and send credit invoice when the barrel is returned; while another sells the barrel outright, agreeing to buy it back any time it is returned in good condition.

The steel barrels used in regular service are generally numbered consecutively by embossing the name and number in the heads, then a record of the movements of these barrels is kept in a card index system. In the ticket system, a red ticket is made out for a barrel leaving the refinery for the station, a white ticket when it is sent to a customer, and a green ticket when it is returned to the refinery. These tickets are mailed to the main office each day and are there placed on pegs on files holding more than 1,000 pegs each. In this way, one company keeps a record of the movements of over 100,000 steel barrels in its service.

Some refiners now use specially designed steel barrels for the distribution of their special products. They thus have distinctive packages which the trade may readily recognize.

In filling stations for steel barrels, the latter are first washed,

[1] See C. E. MILLER, *Oildom*, 5 (1915), No. 2, 29.

and, if any are seriously dented, they are placed in a hydraulic press for the removal of indentations; second, they are rolled through hot ovens, where they are thoroughly dried; and third, they are transported to the paint room, if painting is necessary, and then filled.

TABLE XLIX.—WEIGHTS OF STEEL BARRELS FILLED WITH OILS OF VARIOUS GRAVITIES

(Weights based on "Defiance Extra" barrels of 55-gal. capacity, filled 1 gal. out. For "Defiance Special" barrels, add 15 lb.)

| Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel | Gravity Baumé | R. R. weight per barrel |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 16 | 505 | 26 | 475 | 36 | 450 | 46 | 430 | 56 | 410 | 66 | 395 | 76 | 380 |
| 17 | 500 | 27 | 475 | 37 | 450 | 47 | 430 | 57 | 410 | 67 | 390 | 77 | 375 |
| 18 | 500 | 28 | 470 | 38 | 445 | 48 | 425 | 58 | 405 | 68 | 390 | 78 | 375 |
| 19 | 495 | 29 | 470 | 39 | 445 | 49 | 425 | 59 | 405 | 69 | 390 | 79 | 375 |
| 20 | 495 | 30 | 465 | 40 | 445 | 50 | 425 | 60 | 405 | 70 | 385 | 80 | 370 |
| 21 | 490 | 31 | 465 | 41 | 440 | 51 | 420 | 61 | 400 | 71 | 385 | 81 | 370 |
| 22 | 485 | 32 | 460 | 42 | 440 | 52 | 420 | 62 | 400 | 72 | 385 | 82 | 370 |
| 23 | 485 | 33 | 460 | 43 | 435 | 53 | 415 | 63 | 400 | 73 | 380 | 83 | 370 |
| 24 | 480 | 34 | 455 | 44 | 435 | 54 | 415 | 64 | 395 | 74 | 380 | 84 | 365 |
| 25 | 480 | 35 | 455 | 45 | 430 | 55 | 415 | 65 | 395 | 75 | 380 | 85 | 365 |

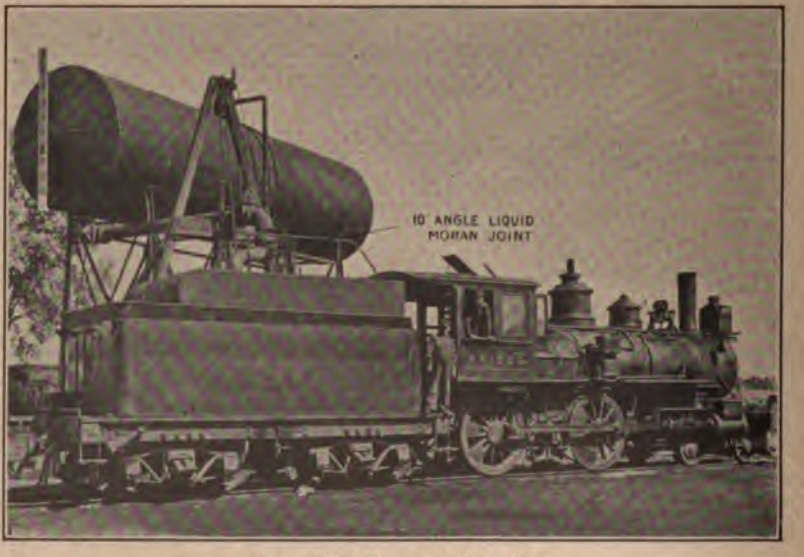



FIG. 175.—Loading fuel oil.



FIG. 176.—The Moran automatic barrel filler.



FIG. 177.—The "Penflex" automatic barrel filler.

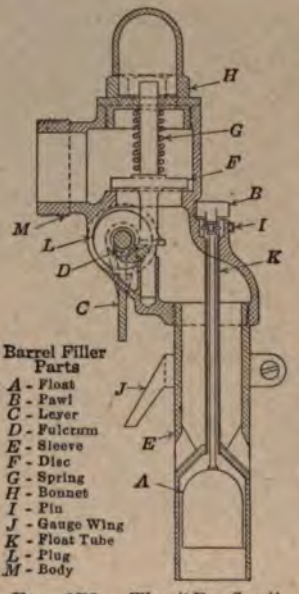



FIG. 178.—The "Penflex" barrel filler.

**Barrel Filling.**—Two types of barrel fillers are shown in Figs. 176 and 177. Fig. 178 gives the construction of the "Penflex" automatic barrel filler, which is, when in use, fitted with 1¼-in. flexible steel hose, as illustrated in Fig. 177.

**Canning.**—The packing of goods in small packages suitable for sale to the consuming trade has also extended to the oil business, and now many jobbers are putting out a full line of all grades of lubricating oils, greases and specialties of all sorts in brightly colored lithographed cans. This business requires considerable warehouse space and detail work, but in many cases has proved very remunerative. One firm is using well over one hundred different styles and sizes of cans.

The canning plant of a prominent eastern company is shown in Fig. 179.



FIG. 179.—View in the canning plant of the Union Petroleum Company, Marcus Hook, Pa.

## SYSTEMATIC METHODS OF TESTING PETROLEUM PRODUCTS

The following scheme of examination of petroleum products is a translation of the tests adopted by the International Petroleum Commission, to which have been added some criticisms of the methods proposed.[1] It simply represents progress in the direction of uniform testing methods.

### ILLUMINATING OILS

I. The *odor* of an illuminating oil is not characteristic as to its illuminating value. If a test (should) be considered necessary, it should be made by shaking (about) 100 c.c. of oil in a bottle of 200 c.c. capacity, with a clear width of neck of 18 mm., at a temperature of about 20°C. for a minute (and then noting the odor.)

II. The test for *color* and *fluorescence* of an illuminating oil is a matter for future agreement. The test for color is to be made with a colorimeter, using standard glasses whose color value is to be determined by comparison with normal liquids yet to be agreed upon. Before testing the color, the petroleum must be filtered through paper.

III. *Specific gravity* is to be determined by the usual methods (officially standardized thermo-areometers, pyknometers, Mohr scales, areometers for small quantities, alcohol flotation process) according to the nature and quantity of the material and the accuracy desired. The standard temperature is fixed at 15°C., the unit of weight to be water at + 4°C., and the specific gravity to be reduced to vacuum. The determination of specific gravity may be made at higher or lower temperature and reduced to the normal temperature at 15°C. by means of coefficients of expansion to be determined by each country for its own petroleum, provided only that the specific gravity does not lie so near a limit that errors could thereby occur. In this case the determination must be carried out at 15°C. or at a temperature very near to this.

IV. In case the determination of *specific viscosity* is desired, it is recommended to use the Engler viscosimeter modified by Ubbelohde for illuminating oils (described in Post, "Chemisch-technische Analyse," **1**, ii, 312).

V. The determination of the *flash-point* of illuminating oils is to be made in the Abel-Pensky apparatus[2].

[1] D. T. Day and F. E. Carter, *Canada Dept. Mines, Mines Branch, Report* No. **291, 1,** 361. On the present practice in the examination of petroleum products, see Kissling, *Petroleum*, **5**, 505, 1009; and Holde's "Examination of Hydrocarbon Oils," trans. by Mueller, **1915.**

For methods of analysis of petroleum itself, see pp. 124 and 127.

[2] The following list of state illuminating oil inspection tests has been compiled by C. D. Chamberlain, Counsel, National Petroleum Association. (See table on following page.)

TABLE L.—STATE ILLUMINATING OIL INSPECTION TESTS

| States | Flash test | Fire test | Instrument |
|---|---|---|---|
| Alabama | ..... | 120°F. | Tagliabue |
| Arizona | No inspection laws | | |
| Arkansas | ..... | 150°F. | Tagliabue |
| California | 110°F. | ..... | Open test |
| Colorado | ..... | 110°F. | Tagliabue |
| Connecticut | 110°F. | 140°F. | Tagliabue |
| Delaware | ..... | 115°F. | Tagliabue |
| Florida | No inspection laws | | |
| Georgia | 100°F. | ..... | Elliott |
| Idaho | ..... | 120°F. | Tagliabue |
| Indiana | 120°F. | 140°F. | Tagliabue |
| Illinois | ..... | 150°F. | Tagliabue |
| Iowa | 100°F. | ..... | None prescribed |
| Kansas | 110°F. | ..... | Foster |
| Kentucky | ..... | 130°F. | Tagliabue |
| Louisiana | 125°F. | ..... | Tagliabue |
| Maine | ..... | 120°F. | Tagliabue |
| Maryland | ..... | 110°F. | Tagliabue |
| Massachusetts | 100°F. | 110°F. | |
| Michigan | 120°F. | ..... | Foster |
| Minnesota | ..... | 120°F. | Tagliabue |
| Mississippi | No inspection laws | | |
| Missouri | 120°F. | ..... | Tagliabue |
| Montana | 110°F. | ..... | Foster |
| Nebraska | 112°F. | ..... | Foster |
| Nevada | No inspection laws | | |
| New Hampshire | 100°F. | 120°F. | Tagliabue |
| New Mexico | ..... | 120°F. | Tagliabue |
| New Jersey | 100°F. | ..... | Elliott |
| New York | 100°F. | 110°F. | Tagliabue |
| North Carolina | 100°F. | ..... | Elliott |
| North Dakota | 100°F. | 125°F. | Elliott |
| Ohio | 120°F. | ..... | Foster |
| Oklahoma | 115°F. | ..... | Tagliabue |
| Oregon | ..... | 120°F. | Tagliabue |
| Pennsylvania | ..... | 110°F. | Tagliabue |
| Rhode Island | 110°F. | ..... | Tagliabue |
| South Carolina | 110°F. | ..... | Elliott |
| South Dakota | 105°F. | ..... | Tagliabue |
| Tennessee | 120°F. | ..... | Tagliabue |
| Texas | No inspection laws | | |
| Utah | ..... | 110°F. | Tagliabue |
| Vermont | ..... | 110°F. | Tagliabue |
| Virginia | No inspection laws | | |
| Washington | ..... | 120°F. | Tagliabue |
| West Virginia | No inspection laws | | |
| Wisconsin | 105°F. | 125°F. | Tagliabue |
| Wyoming | 110°F. | ..... | Foster |

Exportation and transport regulations in the different countries are postponed for further deliberation.

The rules for the determination of the flash-point of illuminating oils, whose flash-point is higher than 50°C., will be based on experiments still to be carried out.

VI. *Cold Test.*—Fresh samples must always be used, and not such as have been previously cooled off to a lower temperature for any considerable time. Testing is necessary only when precipitation might be harmful in the use of an illuminating oil. If, on cooling a sample in the test-glass, precipitation or solidification is observed, then the determination of the cold test must be carried out according to the method recommended for lubricating oils. The freezing point of the distillation residue left from illuminating oils at 275°C. may also be determined. Due regard is also to be given to changes noted during the cooling.

VII. The *fractional distillation* is effected in the Engler bulb according to the continuous method (Ubbelohde, *Mitteilung aus dem Königlichen Material-prüfungsamt*, **1907**, 261; and *Moniteur du Pétrole*, **1908**, 280 and 282). The height of the barometer is to be given and, by making a test with a thermometer of equal dimensions, a correction for the emergent mercury filament to be made. The condenser tube must be entirely dry. The boiling point is that point at which the first drop falls off from the exit tube of the Engler bulb.[1]

The limits of temperature within which the distillates are taken off must be divisible without remainder by 25.

Ordinarily, the fractions are measured volumetrically, the residue, boiling above 300°C. and remaining in the retort, being weighed. For more exact investigations, the weight of the distillates and of the quantity used is determined.[2]

VIII. *Illuminating Power.*—As the unit of measurement of illuminating power, the Hefner amyl acetate lamp is used. For changing from this unit into other units, the tables published by the "Verein der Gas

| Unit | H. | V. | E. | P. | °C. |
|---|---|---|---|---|---|
| Hefner | 1.0 | 0.833 | 0.877 | 0.091 | 0.092 |
| Vereins | 1.2 | 1.0 | 1.05 | 0.109 | 0.111 |
| English | 1.14 | 0.95 | 1.0 | 0.104 | 0.105 |
| 10 c.p. pentane lamp | 11.0 | 9.16 | 9.6 | 1.0 | 1.01 |
| Carcel | 10.80 | 9.0 | 9.5 | 0.98 | 1.0 |

[1] It is better to take the temperature when the first drop falls from the condenser, because frequently this occurs before a drop falls from the exit tube of the distilling bulb.

[2] For the results of tests, including fractional distillations, on American, Bohemian, Galician, German, Rumanian and Russian common and safety illuminating oils, see R. JUNGKUNZ, *Chem.-Ztg.*, **39** (1915), 641 and 659.

und Wasserfachmänner" are used. For exact photometric determinations the grease spot is not appropriate. It is necessary to use the photometer of Brodhun, or Lummer, or other modern apparatus.

The illuminating power of an illuminating oil does not depend solely on its composition, but most particularly upon the construction of the lamp and (the) chimney used to determine the illuminating power. For these tests (of illuminating power), the following points must be observed:

The size of the burner, of the chimney, and the distance from the edge of the burner to the level of the coil before and after the experiment must be given.

Before the experiment, dry the wick (for 2 hr.) at 100°C. (212°F.), then plunge it at once, warm, into the lamp oil, and let it soak there for an hour. The top of the wick should be trimmed with care by means of scissors until an even flame is obtained.

The duration of the combustion should be in general 6 hr. In particular cases, an appreciably longer duration of combustion may be used.

During the first quarter of an hour, turn up the flame to the highest point possible. Later, a quarter of an hour before the first photometric measurement, turn it up again, then leave it to itself (without touching it).

The photometric measurements should be done after the first, second, third, fourth, fifth, and sixth hours.

In determining the total consumption of oil, weigh the lamp before and after the test.

In order to obtain more exact determinations, the lamp should be weighed at each photometric determination. Differences in the temperature of the oil are without sensible effect upon the weight. In addition to the mean illuminating power and the total quantity of oil burned, the consumption per candlepower per hour is to be given. The kind of wick used must be given. The International Commission has left this subject to the study of a special commission.

IX. *Behavior of Burning.*—This subject also has been left to subsequent study by the commission.

X. *Degree of Refining.*

*Acid Content.*—1. Shake 100 c.c. of illuminating oil with 10 c.c. of distilled water with addition of a few drops of an aqueous solution of methyl orange, 1 :1,000. The water must not become pink.

2. Dissolve 100 c.c. of the illuminating oil in 100 c.c. of a freshly neutralized mixture of 4 parts of ether, 1 part of 95 per cent. alcohol, and 1 drop of phenolphthalein solution. Add a drop of 1/10 normal sodium hydroxide solution, and shake in a cylinder with a stopper. If the illuminating oil is neutral, the pink coloring does not disappear during the shaking.

If an acid reaction has been detected by either of the two qualitative

tests, then the quantitative determination of the acid content is to be undertaken by the known analytical methods. Meanwhile, it may be of value to test the behavior toward sodium hydroxide by using the so-called "soda test" (Muspratt, "Technische Chemie," **1898,** 234; or Post, "Chem. Tech. Analyse," **1,** 320), in which sodium hydroxide solution (1.02 sp. gr.), diluted with water in ratio 6:100, is used. However, the result of the soda test alone is not decisive; but when it discloses nothing, a possible acid content or "acid figure" is to be determined by 1 or 2, or, if no free acid is present, the ash content (see XII).

X*a*. *Hydrocarbon Characteristics.*—The determination of hydrocarbon content is referred to a committee for further study.[1] The problem of this committee is to be the preparation of methods for determining the various hydrocarbon groups that go to make up petroleum and its products.[2]

XI. *Storage Stability.*—For studying the liability to alteration of

[1] Nominated as members of special committee are ENGLER, HOLDE, ZALOZIECKI, EDELEANU, GURWITSCH, NICOLARDOT, ALLEN, and SLUYTERMAN VAN LOO.

[2] In this regard, ENGLER has communicated the following partial method worked out in his laboratory by TAUSZ and PFEIFFER: For qualitative detection of the unsaturated hydrocarbons, a small quantity of benzine is shaken and boiled with concentrated aqueous mercuric acetate solution. Olefines and cyclic unsaturated hydrocarbons dissolve cold and are oxidized in boiling. In this process, they reduce the mercuric acetate, which precipitates on cooling. Moreover, butylene colors red, amylene yellow, and hexylene pink. For their quantitative determination, the unsaturated compounds are for the most part thoroughly shaken with mercuric acetate at ordinary temperature and distilled out of the solution after adding hydrochloric acid. These regenerated unsaturated hydrocarbons are identified as formolite in the usual way in which the cyclic ones are identified. The remainder of the unsaturated hydrocarbons of the benzine, not dissolved by mercuric acetate, is destroyed (decomposed) with fresh mercuric acetate by boiling with a return condenser, whereupon the remaining more stable hydrocarbons are distilled off, separated by a special method, and determined quantitatively. The difference between the volume of the benzine used and that of the last-named distillate gives the content in unsaturated hydrocarbons. The correction for unavoidable losses is found by a blank experiment.

The subsequent analysis is qualitative. The aromatic hydrocarbons are detected as formolite. In their presence, the formolite reaction with the total distillate is repeated, until the hydrocarbons distilled off no longer give formolite. For detecting the cyclo-hexanes in this remainder, a sample is passed at about 300°C. in a hydrogen atmosphere over finely divided nickel. If the condensate now shows the formolite reaction, then there were cyclohexanes present. ZELINSKI thinks they can be determined quantitatively by dehydrating with palladium black. Naphthenes and paraffins are recognized as usual from the physical constants of the carefully fractionated remainder.

petroleum in storage, a special committee is named to collect and work up material relating to this question for presentation at the next meeting of the International Commission.[1]

XII. *Ash Content.*—For determining the ash content, about 1 liter of filtered oil will be used, taking into consideration the filter residue qualitatively and, if necessary, quantitatively. The ash must be indicated in percentage by weight. The ash is determined by distilling off the sample to about 30 c.c. This residue is poured off along with the last portions of the distillate into a platinum dish, evaporated to dryness with removal of the vapors, and incinerated.

XIII. *Sulphur Determination.*—The determination of the sulphur is effected quantitatively, either according to the method of Engler-Heusler (*Z. angew. Chem.*, **1895**, 285; Engler, *Chem.-Ztg.*, **1896**, 197; Post, "Chemisch-Techn. Analyse," **1**, ii, 321), making, if necessary, at the same time a blank test to eliminate errors arising from the $SO_2$ content of the air of the laboratory, or else by the method of combustion in the bomb.

XIV. *Water Determination.*—Water determination for illuminating oil is dispensed with. In case of the occurrence of clouding, it must be determined if this results from the water content.

### BENZINE (Gasoline, Naphtha, Etc.)

I. *Specific Gravity.*—Same as for illuminating oils.

II. *Distillation.*—The tests are the same as for illuminating oils. Fractionating is to be effected while making the limits of temperature divisible by 10. Final point of the distillation is to be regarded as that temperature at which the bottom of the retort appears (to be) dry, or when white vapors make their appearance.

III. *Degree of Refining.*—Tests to be omitted until a uniform method is fixed.

IV. *Flash-point.*—In case such test is to be made, German Abel-Pensky apparatus is to be used.

V. *Acid Content.*—The rules given for illuminating oils are to be used, *mutatis mutandis.*

VI. *Heavy Hydrocarbons.*—Referred to the committee on point X*a* for illuminating oils.

VII. *Water Determination.*—The same rules as for illuminating oils.

VIII. *Calorific Value.*—To be determined by bomb or other suitable apparatus. Indication must be given as to which apparatus was used.

IX, X and XI.—(High-boiling constituents, odor and color, and illuminating power.) These were referred to a special committee to investigate and report as in the case of illuminating oils.

[1] SINGER is named as chairman, and the following are members: ALLEN, GUISELIN, GANE, SOHN, and BERGUER.

## LUBRICATING OILS[1]

I. *Color.*—Transparency of the oils in a thin layer is to be determined by letting them run over a glass surface. Color is to be determined, as a rule, by simple ocular inspection in a test tube. In special cases, examination is made in rectangular vessels 10 cm. in height, 10 cm. in length, and 15 mm. in width (inside measurement), made of pure white glass of 5 mm. thickness of wall, and by both transmitted and reflected light. The color is of no influence on the lubricating value, and, as a rule, is regarded only as an assistance for identification.

II. *Specific Gravity.*—(*a*) Limitation of the specific gravity with reference to the proposed use is not necessary, and must not be subjected to too narrow limits. Only when oils of a determined origin are demanded, certain limits of gravity may be set for classification purposes, and these must not be drawn too narrowly. (*b*) Specific gravity serves only as a means of classifying mineral oils of known origin, and for tests of identity and comparison. (*c*) The determination of the specific gravity is effected according to the nature and quantity of the material and the degree of accuracy desired, according to the usual processes (officially standardized areometers, pyknometers, Mohr scales, areometers for small quantities of oil, alcohol flotation process). For thick oils, the method of mixing with kerosene is to be carried out. The standard temperature of 15°C. is fixed; as unit of weight, water at + 4°C.; also specific gravity is always to be reduced to vacuum. The determination of specific gravity may also be effected at higher or lower temperatures than 15°C., and be corrected by the coefficients of expansion to the normal temperature of 15°C. Each country must determine for its own mineral oils the coefficients of expansion (correction figures). In case the specific gravity so calculated lies so near to a limit that errors might arise, the determination must be carried out at 15°C., or the coefficient of expansion (correction) exactly determined.

III. *Flash-point.*—(*a*) In all cases where it is a question of obtaining the greatest possible exactness, the Pensky flash-point apparatus must be used; in other cases also the open cup, preferably with a mechanical device for bringing up the test flame and with an adjustable burner. (*b*) In case of absence of other prescribed methods, a porcelain cup 4 cm. in width and 4 cm. in height is to be used for the determination of the flash-point in the open cup. This is to be set in a sand bath up to the level of the oil. (For mode of experiment, see Holde, "Untersuchung der Mineraiöle und Fette," 3rd ed., 13.) In determining the flash-point in the open cup, a thermometer with a short bulb is to be used, similar to that used with the Pensky apparatus; the center of the bulb must be in the center of the oil. Indication must always be given whether the work was prosecuted with or without consideration of

[1] See also STILLMAN'S "Examination of Lubricating Oils," **1914.**

the correction for the emergent stem of the thermometer. The officially standardized thermometers for the Pensky apparatus are so graduated that the indication of error includes the last-mentioned correction (at the same time).

III*a*. *Evaporation.*—Referred to a special committee.

III*b*. *Distillation Test.*—Distillation test is in general to be effected in the Engler bulb, using 100 c.c. Distillation is to be carried on to 270°C., the temperature being taken in the vapor.

IV. *Burning Point.*—This determination is carried out in the open cup, as in the case of the flash-point. The heating must be continual, at the rate of about 4°C. a minute—at the outside, 6°C. a minute.

V. *Specific Viscosity.*—The Commission has referred this subject to a special committee.

VI. *Capillarity.*—Also referred to a special committee.

VII. and VIII. *Cold Test.*—(*a*) For the simple determination the test tube method is sufficient, to be carried out in the following way: Into two test tubes of 18 mm. diameter, the oil is to be poured to the height of 3 cm. In one of these test tubes a thermometer is dipped into the liquid. Both samples are kept for an hour in a freezing mixture at the desired temperature. The sample without the thermometer is then tilted, and the consistency, that is, the flowing capacity of the oil, determined. (*b*) In case of numerical comparison of the flowing test by the U-tube process, the sample in the test tube is to be cooled off for an hour to the experiment temperature, while maintaining 50-mm. water pressure in the tube with width of 6 mm. (limit of error of plus or minus 0.3), pressure action lasting for a minute with a stipulated minimum rise.

(*c*) Preliminary treatment of the samples: For the consideration of the changes in the cold point conditioned by the influence of temperature changes, the samples are to be tested in two separate experiments, in the condition when delivered and after heating for 10 min. to 50°C. In the case of the oil tested in the heated condition, the determination is to be repeated for safety if the oil (left over) from the first test is sufficient. (*d*) For removing chance impurities, the oil before heating is to be poured through a sieve with a ⅓-mm. mesh. For this purpose very thick oils must be heated slightly. (*e*) Oil containing water must be decanted, and afterward filtered through cotton wool dried at 100°C.

IX. *Asphalt-like Bodies.*—Referred to special committee.

X. *Water and Mechanical Admixtures.*

(*a*) *Water.*—The water content of oils is to be determined quantitatively, only in case the qualitative test has shown a notable water content. In case of pure mineral oils, which, at 50°C., have an Engler degree of less than 8, the determination is made in the following way: About equal weights (each about 100 grams) of the original and of the dehydrated oil are heated in glass dishes on a boiling water bath until the formation of foam (scum) has ceased, and the loss in weight determined. From the

difference in loss of weight of the two samples the water content in the original oil is to be calculated. The dehydration of the oil, before heating, is effected by shaking the slightly heated oil in an Erlenmeyer flask with calcium chloride, and afterward filtering on a dry filter. In case of oils of more than 8 Engler degrees, it is sufficient to establish the loss of weight by heating, and a parallel experiment with dehydrated oil is superfluous. In other cases, for instance with oil of high water content (solid fats), the determination according to Marcusson is to be employed, depending on the distillation of a considerable quantity of oil with xylol and the measurement of the water distilled over. All the other usual methods of testing (usual) for this purpose can be used, but the process must always be specified.

(*b*) *Mechanical Impurities.*—5 to 10 grams of well-shaken oil are dissolved in a glass cylinder in 100 c.c. of benzine (in case of light colored oil from which no asphalt will separate, benzine may be used also). After standing all night, the solution is poured through a weighed filter. The cylinder is well rinsed out and the filter washed with the solvent until the wash liquid after evaporating on the water bath no longer gives a residue. The filter is dried at 105°C. to a constant weight and then weighed.

XI. *Ash*, XII. and XIII. (*Stability to Heat and Air in Storage*), and XIV., XV. and XVI. (*Stability toward Water Vapor, Superheated Steam and High Pressure*).—Deferred for further investigation.

XVII. *Degree of Refining.*—Free Acids:

(*a*) Existing processes for determining free acids in lubricating oils are retained. Mineral acids are to be determined in the aqueous extract of about 100 grams of oil, with the use of methyl orange as indicator as in the case of illuminating oil. Organic acids are to be determined by titration with alcoholic $\frac{1}{10}$ normal caustic alkali; an alcohol-ether mixture is used to dissolve 10 c.c. of a light oil, and in the case of a dark oil absolute alcohol is the solvent. (*b*) The quantity of mineral acid is to be calculated in percentage of $SO_3$; the organic acids expressed as "acid figure." (The acid figure indicates the number of milligrams of KOH necessary to saturate the free acids contained in 1 gram of oil.)

Free Alkali.—Determine the presence of alkali in the aqueous extract of at least 100 grams of oil.

Salts (Ash Content).—Inorganic salts are determined in an aqueous extract of 100 c.c. of oil. The presence of alkali soaps in case of pure mineral oils generally makes itself manifest by permanent emulsion and slightly alkaline reaction of the aqueous extract, in which case the qualitative detection of the alkali soap is effected by the following method: In a test tube of 15 mm. diameter 5 c.c. of 0.5 Baumé caustic alkali solution is heated to boiling over the Bunsen burner. An equal quantity of oil is added and it is heated again for a minute to boiling, in such a way that the two liquids mix as intimately as possible during the boiling.

This sample is then set for 2 or 3 hr. in a boiling water bath. Then the sample, on inspection, must show the following results: The oil, in case it is pure, must be clear, and the alkali extract must appear transparent in so far that small print can be read through it. Clouding indicates naphthenic acid salts, and in that case the ash determination must be carried out.

XIX. *Admixtures.*—(*a*) Fatty Oil: Fatty oil is detected qualitatively by the heating for a quarter of an hour of 3 or 4 c.c. of the oil to be tested in a paraffin bath (oil bath) at about 340°C. with a small piece of sodium hydroxide or, in doubtful cases, with metallic sodium After cooling off to ordinary temperature, the oils show some lather in case of presence of fatty oils, or else they gelatinize. The soap lather is in the case of cylinder oils the deciding sign of the presence of fatty oils. Naphthenic acid may produce similar phenomena. Quantitatively, fatty oil is determined according to the approximate quantity and the degree of exactness desired, by determining the saponification figure or gravimetrically according to Spitz and Hönig.

XX. *Paraffin.*—The determination of the paraffin content can generally be dispensed with in testing lubricating oils because any high percentage of paraffin would become apparent during the cold testing. In special cases, for instance in litigation tests, etc., the alcohol-ether process of Engler and Holde for paraffin determination can be used.

XXI. *Lubricating Capacity.*—This point, as well as the study of transformer oils, sampling and measuring mineral oils kept on supply in tanks, ships, and so forth, is deferred.

## CHAPTER XIII

## SPECIAL REFINERY TECHNOLOGY

This chapter, which supplements the technology presented in Chap. XII, comprises brief considerations of certain special refinery operations and a rather detailed account of the chemical treatment of petroleum distillates. Many of the processes referred to are of little or no technical importance, but they have been included to show the trend of inquiry and invention.

### THE PROCESSES OF "SUNNING" AND "REDUCING"

Paraffin-base petroleums which were not sufficiently heavy *per se* for use as lubricants, but were too heavy to be worth distilling for the production of illuminating oil, were at one time worked up to some extent by the processes of "sunning" and "reducing."[1]

In the "sunning" process, the petroleum was placed to a thickness of about an inch upon a layer of water, in a wooden tank sunk in the ground, and having a length of 60 to 70 ft., a width of 20 to 30 ft., and a depth of 1 ft. The water was heated by a horizontal steam coil to about 110°F., so that foreign matter in suspension was deposited. In a few days the density of the oil increased from 32° to 29°Bé., with a loss of about 12 per cent. of the volume.

The original "reducing" process consisted in the partial distillation of the petroleum, by the careful application of heat, so as to bring it up to the required viscosity, what were regarded as superior lubricating oils being thus obtained. In one method the petroleum was distributed in thin streams over sheets of loosely woven cloth suspended above troughs in a heated chamber. As the oil ran down into the troughs, any solid particles present were retained by the cloths, while the lighter portions of the oil evaporated, so that a bright green odorless oil, said to be of high value as a lubricant, was produced.

[1] Cf. p. 452.

The method of reduction principally adopted consisted in partial evaporation in stills, either solely by direct heat or with the aid of steam, and sometimes *in vacuo*. Anything approaching destructive distillation or "cracking" was carefully avoided, as it would result in the reduction of the viscosity of the product. Only the benzine and a sufficient quantity of the heavier oils were run off to bring the residue to the required density, which was 29° to 32°Bé. for oil for large journals or those revolving at moderate speeds, or 26° to 29°Bé. for small, high-speed journals or for the interior of steam-engine cylinders. Peckham[1] stated that this process was used for a large proportion of the lighter-grade oils of West Virginia and Ohio, and for the whole product of the Smith's Ferry district. To quote him: "The latter oil is very peculiar, having the color of pale sherry, without its transparency, and when freshly pumped has a gravity of 50°Bé., with a much less pronounced and less disagreeable odor than any other petroleum produced in commercial quantities in the United States. When reduced with the aid of steam, the distillate of suitable specific gravity for burning oil requires little or no treatment with acid or alkali, and the 'reduced' oil from the still preserves its amber color and freedom from offensive odor, furnishing a lubricator of very superior quality and attractive appearance."

### THE DEHYDRATION OF PETROLEUM[2]

A number of processes have been patented for effecting the dehydration of petroleum. To cite some examples: Münster[3] heats petroleum above the boiling point of water and squirts the heated oil under pressure against a turbine wheel arranged in a tube enlarged upward in box form, the mass flowing down being worked by stirrers operated by the turbine so that a product claimed to be anhydrous flows down continuously; Dittersdorf[4] destroys the surface tension of the emulsion by the mechanical action of sharp-angled materials, such as sharp sand, iron turnings, etc., the emulsion being mixed with the sharp materials in

[1] "Report on the Production, Technology, and Uses of Petroleum and Its Products" (10th Census), *Quart. Rept. U. S. Bureau of Statistics*, **10** (1888).

[2] On the dehydration of tar for spraying, macadam and pitch grouting, see CHAMBERS, *J. Gas Ltg.*, **132**, 261; and WARDELL, *ibid.*, 375.

[3] German Patent 231222, Mar. 23, 1910.

[4] Austrian Patent 2229–11, Mar. 14, 1911.

a heated filter by means of air or a stirring device; and Stone[1] heats a stationary body of petroleum sufficiently to cause the free water to settle out, draws off the oil, and then causes it to flow rapidly over a metallic surface heated above the boiling point of water, in order to break up the emulsion. Then, too, Graaff[2] has worked out a process for the separation of water from petroleum by heating; and the Dampfkessel- und Gasometer-Fabrik A.-G. vorm A. Wilke & Co.[3] has patented a special apparatus for the removal of water and salts from crude mineral oil.

The electrical process of Cottrell and Speed has received some attention.[4] In this process, after the water that separates out from the petroleum by gravity is drawn off, the oil is pumped into vertical cylinders of special form, 3 ft. in diameter and 12 ft. high, fitted inside and outside with specially constructed electrodes. A current of 10,000 to 15,000 volts is then passed through the oil, short-circuits being prevented by rotation of the electrodes. It is said that the minute drops of water, as conductors of the current, arrange themselves chain-like from electrode to electrode along the electrostatic lines and form larger drops which sink to the bottom of the cylinders.[5] It has been reported[6] that the total operating cost of this process is $0.015 per barrel of petroleum treated and that a 2-treater plant will handle 1,000 bbl. per day.

Laird and Raney[7] claim to break up emulsions of petroleum and water by passing the liquid upwardly between electrodes and supplying a high potential to the electrodes to coalesce the particles of water by the action of heavy short-circuit currents; the electric current is interrupted intermittently to allow the water to form into large free globules. The same inventors have also patented an apparatus for breaking up emulsions of petroleum with water, electrolytically.[8]

In 1912, the appearance of water in the crude oil of the Nevada

[1] United States Patent 1070555, Aug. 19, 1913.

[2] German Patent 248872, Mar. 25, 1910.

[3] German Patent 244064, Jan. 20, 1911.

[4] See Beazley, *Oil Age*, 1911, 21; also, *Petrol. Rev.*, 26, 284; *Trans. Am. Inst. Min. Eng.*, 43, 514; and *Western Eng.*, April, 1912.

[5] All salts are said to be removed along with the water

[6] *Petrol. Rev.*, 26, 284.

[7] United States Patent 1116299, Nov. 3, 1914.

[8] United States Patents 1142759-60-1, June 8, 1915.

Petroleum Company, of Coalinga, Cal., rendered necessary the installation of a dehydrating plant to reduce the water below the 3 per cent. limit prescribed by the agency. Hardison[1] has described the experiments which were made in order to determine the most satisfactory plan for this purpose.

The water in Coalinga petroleum occurs both free and as an emulsion. Free water settles out readily, but the emulsion requires special treatment: since it consists of globules of water enveloped by an oil film, it can only be broken up by heating the water to at least its boiling point, when an explosion occurs, destroying the globules.[2] After preliminary experimentation,

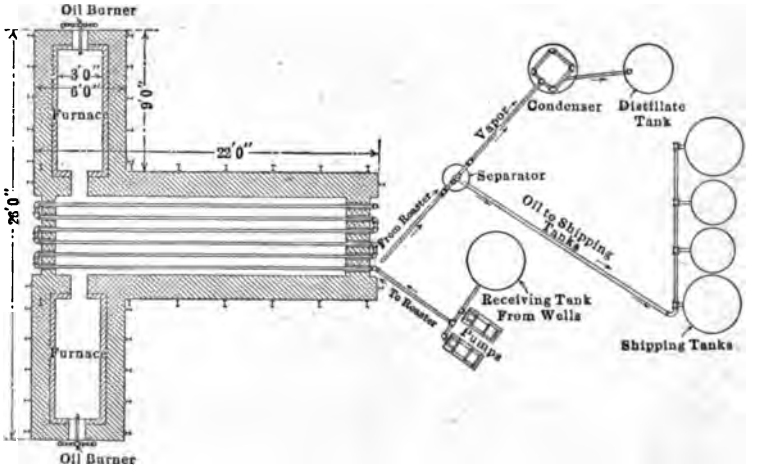



FIG. 180.—Plan of the oil-dehydrating plant of the Nevada Petroleum Company, Coalinga, Cal.

Hardison states that it was concluded that exhaust-steam coils could not be used successfully for heating the oil because the temperature was not raised sufficiently high, and that the temperature must be raised to a point which would generate steam, thereby liberating the water and causing it to separate from the oil. Accordingly, a plant was erected, consisting of brick walls lined with firebrick, inside of which were coils of 3-in. pipe, through which the oil was pumped. These coils were constructed in horizontal rows of five and six 20-ft. joints below the

[1] *Bull. Am. Inst. Min. Eng.*, **1915**, No. **99**, 637. On the dehydration of California petroleum, see also BEAZLEY, *Western Eng.*, **1** (1912), **56.**

[2] For a consideration of water emulsions in petroleum, see **WRIGHT,** *Western Eng.*, **1** (1912), 307.

arch and nine joints in each of the three rows above, 54 joints in all. Furnaces were built on either side of the front, so that the flames would not come into direct contact with the coil. The heat from the furnace passes into the lower compartment, thence to the back and under the arch, then through the opening at the back, into the top compartment and out through the stack at the front end. A receiver was erected for the oil as it left the roaster, the function of which is to separate the vapor from the oil and to recover the light products that would otherwise pass off into the atmosphere and be wasted. The oil passes out at the bottom of this receiver and into the shipping tanks; the vapor passes out of the top through a coil submerged in cold water,

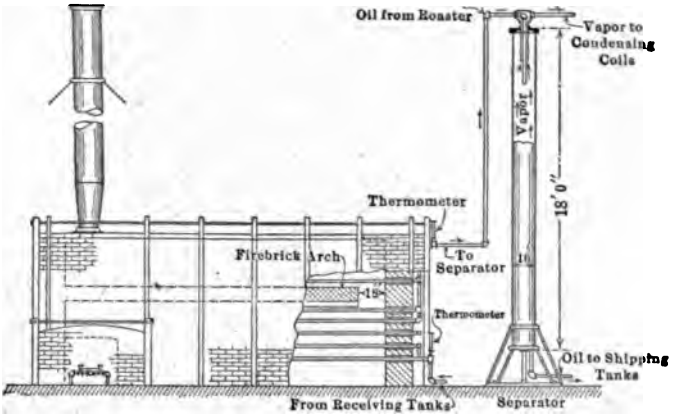



FIG. 181.—Side elevation of oil-dehydrating plant.

where it is condensed and from which it is allowed to collect in a receiving tank.

This plant, illustrated in Figs. 180 and 181, has proved a complete success. Because the coils are small, the solids from the oil cannot precipitate and form blisters. It was operated daily during 1914 without any repairs.

The cost of operating the dehydrating plant per 100 bbl. treated is as follows: Labor, 75 cts.; fuel consumed, 74 cts.; steam consumed, 22 cts.; total, $1.71. The condensation per 100 bbl. is: Water, 10.40; light oil, 0.43; total, 10.83. The gravity of the light oil is 37.2°Bé. The oil before treating showed: Temperature, 80°F.; gravity, 14.8°Bé.; B. S. and M., 11.6 per cent. The oil after treatment showed: Temperature, 260°F.; gravity, 15.5°Bé.; B. S. and M., 1.0 per cent.

When water is present in a free state in California petroleum, it is sometimes separated by piping steam into a storage tank in 1- or 2-in. coils, placed horizontally 4 to 6 in. from the bottom; a temperature of 100° to 150°F. is generally sufficient to cause the water to settle to the bottom, where it is drawn from the tank by a valve placed for the purpose.[1] If, however, the oil is emulsified, one of four systems is usually used: (1) the Cottrell process, described above; (2) dehydration by direct heat; (3) dehydration by indirect heat; or (4) dehydration by compressed air.

Most of the methods of dehydrating by direct heat have no provision for preventing loss by evaporation, although one patented system is said to overcome this objection.[1] If the emulsion is not too refractory, the oil may be dehydrated by indirect heat in the following manner: It is pumped into the bottom of an 8-ft. by 20-ft. tank through a spider with, say, $\frac{1}{12}$-in. holes. About 500 ft. of 2-in. pipe are used for a steam coil and at least 10 ft. of water is kept in the tank. The water is maintained at a temperature of from 150° to 200°F., and serves to separate the oil and water as the petroleum to be dehydrated goes through it. The oil rises to the top, and water may be run off whenever necessary, in order to keep a level of about 10 ft.

The Milliff dehydrating system has been used with success in the Pacific Coast fields. An air pressure sufficient to overcome the weight of the petroleum is maintained by an air compressor through a 3-in. line which passes under a boiler furnace, at which it is heated to a temperature of 1,000°F. The heated air is conveyed through an insulated line to a tank 8 ft. in diameter and 20 ft. high, which it enters at the base through a spider with four 3-in. wings having $\frac{1}{16}$-in. holes and intermingles with the oil. The water in the petroleum is converted into steam by the heated air and simultaneously the oil is liberated from the emulsion. It has been reported that one set of heater pipes in the boiler furnace served to clean 140,000 bbl. of petroleum at the Port Costa pumping station of the Associated Pipe Line; the oil contained an emulsion of 30 to 60 per cent. and tested less than 1 per cent. after treatment by the Milliff system.

[1] PAINE and STROUD'S "Oil Production Methods," **1913, 163.**

[1] *Ibid.*, 167.

## THE CONTINUOUS DISTILLATION OF PETROLEUM

A number of processes have been devised for the continuous distillation of petroleum, in which oil is supplied to the stills as fast as the distillate passes off.[1] This system was first put into extensive practical use in Russia,[2] where it is now largely employed, and it is in use by a number of American refiners,[3] particularly of the Gulf[4] and California[5] fields.

It is understood that the continuous system is largely made use of in the refineries of the Gulf Refining Company and of the Texas Company, not only for crude running and steam-stilling, but also for the distillation of lubricating oils. While it has been maintained that continuous operations do not permit of sufficient cracking to obtain the highest yields of light products, there are really few instances in petroleum refining where cracking is a *quality* argument. Refiners handling large quantities of petroleum are adopting more generally the continuous system, for this method has practically all the advantages of economy, quality, and much greater capacity, and, in addition, lends itself to a much better regulation of condensing equipment and uniform separation of the distillates.

The continuous system was first applied generally to the fire-stilling of American petroleum in 1899, by the Atlantic Refining Company, of Philadelphia, Pa., although certain other companies had employed the method in steam-stilling prior to that time. The apparatus for continuously distilling petroleum, patented by Max Livingston,[6] of Philadelphia, Pa., was perfected and was in operation in 1899, at the plant of the Atlantic Refining

[1] On the continuous distillation of petroleum, see especially FUHST, *Dingler's polyt. J.*, **207** (1873), 293; GASTER, *Petrol. Rev.*, **1** (1899), 465; VON GRÖLLING, *idem*, **2** (1900), 130; JACOBI, *Dingler's polyt. J.*, **159** (1861), 150; and ROSENTHAL, *Chem.-Ztg.*, **34** (1910), 1278. See also p. 455.

[2] See this section, p. 550.

[3] The employment of the continuous system has been frequently attempted in the Appalachian field, but it is said that many of the results have been unsatisfactory because of the comparatively large amount of dissolved gas contained in the petroleum, the high yield of illuminating oil, and the less fluid characters of the residues.

[4] It may be noted here that the petroleum of the Gulf field possesses some of the characteristics of Baku petroleum.

[5] In 1915, by the American Petroleum Company, the Los Angeles Oil Refining Company, and the Union Oil Company (see pp. 506, 508 and 509.

[6] United States Patent 728257, May 19, **1903.**

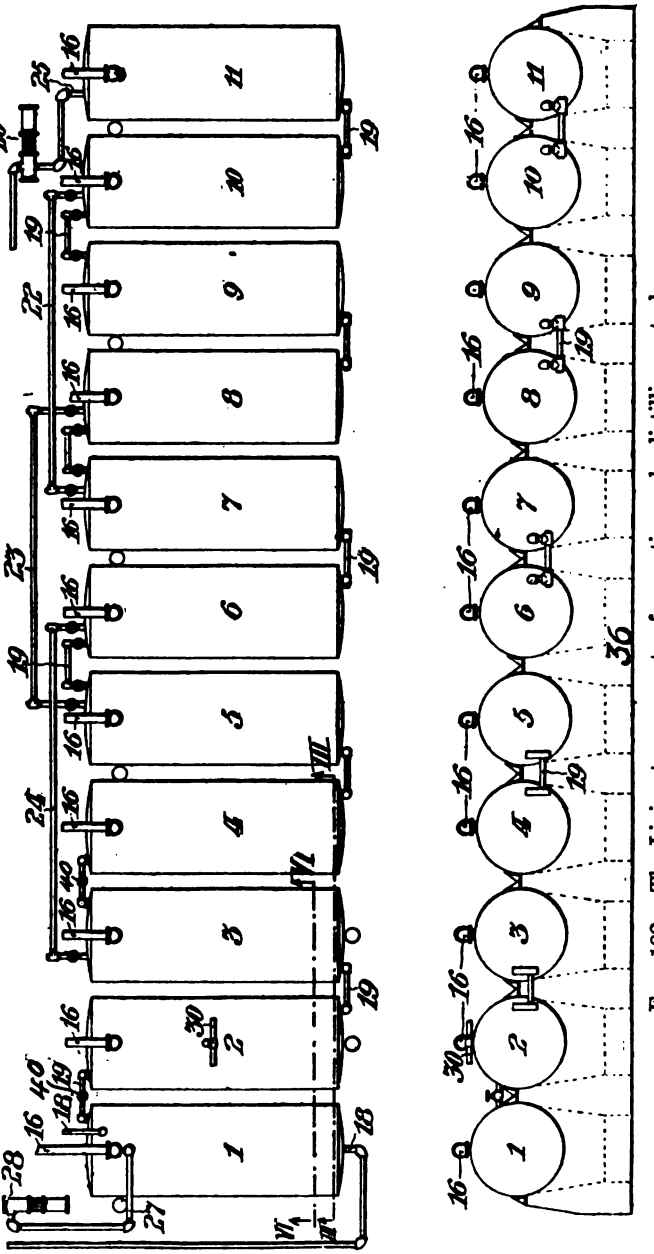



FIG. 182.—The Livingston apparatus for continuously distilling petroleum.

*Upper.*—A view in top plan of a battery of stills arranged in accordance with a good form of Livingston's invention.
*Lower.*—A view in front elevation of the stills shown above.

Company in Philadelphia, and all continuous distillation installations now in use in the United States are said to be based upon the form of plant embodying this invention.

**The Livingston Apparatus.**—Livingston's invention relates to distilling apparatus and its object is the provision of a simple, safe, economical, and efficient apparatus in which the distillation of crude petroleum may be carried on as a continuous operation.

In the accompanying illustrations, Fig. 182 (*upper*) is a view in top plan of a battery of stills arranged in accordance with a good form of this invention. Fig. 182 (*lower*) is a view in front elevation of the stills. Fig. 183 is a view in elevation of a series of stills, the illustration being partly in section and on an enlarged scale as compared with Fig. 182. Fig. 184 (*upper*) is a

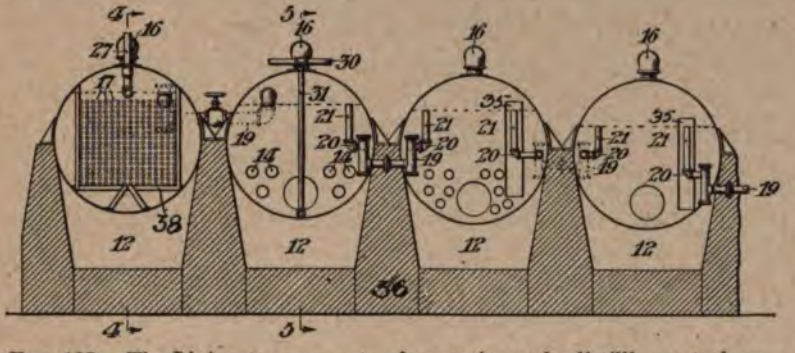



FIG. 183.—The Livingston apparatus for continuously distilling petroleum.
A view in elevation of a series of stills, the illustration being partly in section and on an enlarged scale as compared with the preceding figure.

sectional elevation of the preparatory still 1, shown in Fig. 183, section being on the line 4–4 of Fig. 183. Fig. 184 (*lower*) is a sectional elevation of one of the vaporizing stills, for instance, that designated 2, section being supposed on the line 5–5 of Fig. 183. Fig. 185 (*upper*) is a view of several stills, illustrating a modified form of short connection. Fig. 185 (*lower*) is a detail of one of said short connections. Similar letters of reference indicate corresponding parts.

In the initial distillation of the crude petroleum under the usual practice, the operation is carried on until all the lighter oils have been vaporized, leaving the residuum to be subjected to subsequent independent operations. Livingston's invention has especial, although not restrictive, relation to this initial distillation of crude petroleum, and it is sought to enable said distilla-

tion to be carried on in as economical and efficient a manner as possible, producing distillates of standard quality and grade, and at the same time preserving in the residuum as large a proportion as possible of the constituents useful in by-products. The vapors given off in this initial distillation are condensed and conducted to suitable receptacles, according to their character, for further operations. The number of such receptacles and the subdivision or classification of the condensed vapors according to their grades or characteristics, may be extended as far as may be desired, and to such end any required number, large or small, of stills and condensate receptacles may be employed.

In Figs. 182 and 183 are the stills of a series (1 to 11), shown as arranged side by side, on a foundation of masonry 36, which embodies a series of fire chambers 12, one situated beneath each still. Each still, with its fire chamber, is structurally independent of every other, except for the connections hereinafter described. Each still is preferably formed as a cylindrical vessel of sheet iron, and placed upon its side across the top of its associated fire chamber. At the back of the fire chambers of the first two stills of the series after No. 1, are arranged up-takes 13, Fig. 184, discharging into a series of flues, 14, which extend through the bodies of said stills Nos. 2 and 3, and discharge within the smoke boxes 15, each of which is common to the group of flues of a single still, from which boxes lead suitable smoke stacks. The remaining stills of the series may be provided at the backs of the fire chambers with any suitable smoke stacks or other conveyors for the products of combustion, by which the latter are discharged at any suitable point or utilized in any desired manner—the flues 14, as stated, not being ordinarily used in such remaining stills of the series. The passage of the smoke and products of combustion through the flues within the bodies of the stills Nos. 2 and 3, of course, aids in elevating the temperature of the oil in said stills and thus economizes fuel. 16 are vapor pipes leading from the stills, and through which the vapors or distillates are conducted to suitable condensers and collecting tanks (not shown). Still No. 1, which is termed the preparatory still, is provided with an internal steam or other heating coil 17 (see Fig. 184), the supply and discharge pipes 18 of which pass through the shell of the still, and through which coil is charged steam or hot fluid from any suitable source. In practice said coil 17 may be supplied with exhaust steam.

Said coil 17 may, of course, be arranged in any desired manner. It is preferably arranged with its pipes separated from each other by suitable interspaces to allow the free precipitation of earthy matter contained in the oil, and may be supported by any suitable framework, such as shown in Fig. 183.

Referring now to the pipe connections, and especially to the

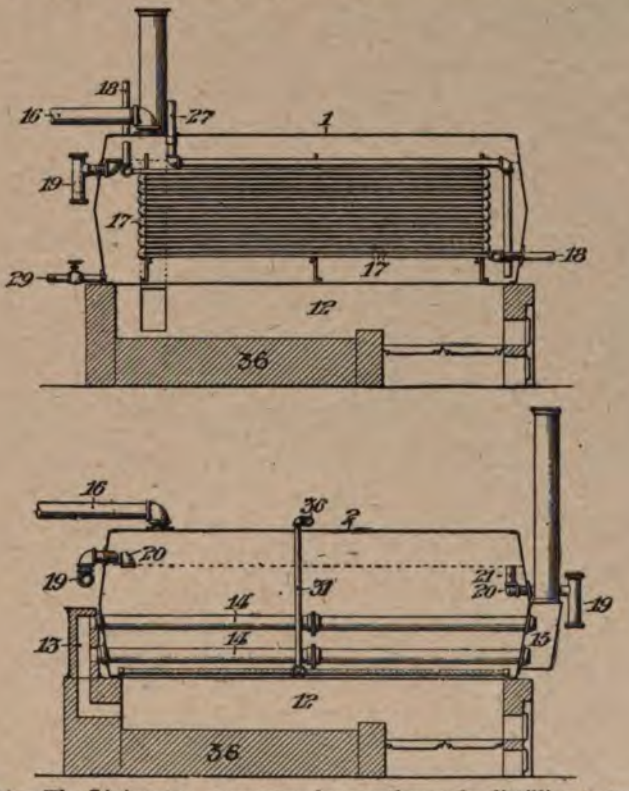



FIG. 184.—The Livingston apparatus for continuously distilling petroleum.

*Upper.*—A sectional elevation of the preparatory still 1.
*Lower.*—A sectional elevation of one of the vaporizing stills, for instance, that designated 2.

construction shown in Figs. 182, 183 and 184, each pipe connection connective of two adjoining stills, and which connections are herein termed the short connections, is as to the portion of it which exists outside of the stills, of approximately U-shaped form, to constitute a suitable trap, such U-shaped portion being designated 19; and from the respective side members of such portion 19, branches lead to the interior of the two

stills connected, such branches (especially the inlet or receiving branches, that is to say, the branches into which the oil flows as it enters the short connections) being provided within the respective stills with elbows 20, to which regulating sections 21 may be fitted and secured. As indicated in the several figures, said regulating sections 21 are in screw threaded connection with the pipe elbows 20, from which they may be unscrewed as desired. The short connections located at the rear of the stills, to wit, the ends thereof appearing in plain view at the upper edge of Fig. 182, are preferably provided with cocks or valves, by which the flow of oil through them may be shut off

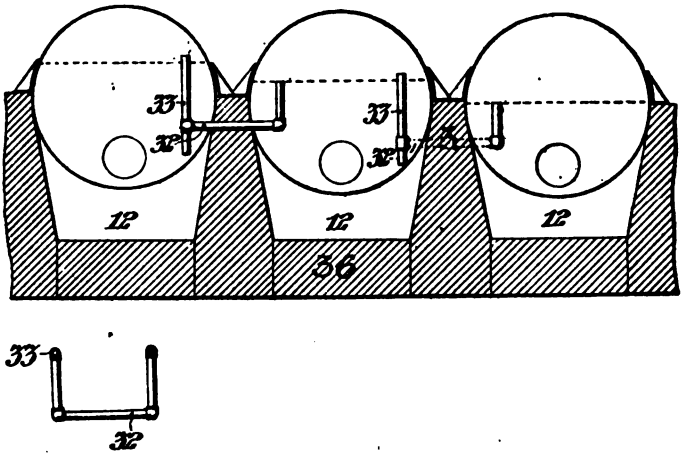



FIG. 185.—The Livingston apparatus for continuously distilling petroleum.
*Upper.*—A view of several stills, illustrating a modified form of short connection.
*Lower.*—A detail of one of these short connections.

when desired. Those located at the front ends of the stills are preferably not so provided. 22, 23 and 24 are long connections, arranged, in the embodiment illustrated, to connect respectively stills 7 and 10, 5 and 8, and 3 and 6. Except for being proportionately longer, the long connections are shown as of substantially the same construction as the short connections described. 26 is a pump connected by the pipe 25 with the last still of the series and adapted to pump residuum from said still. 27 is an oil supply pipe, connected with a pump 28, by which the petroleum is charged to the preparatory still. 30 is a steam supply pipe having a branch 31 leading to the bottom of still No. 2, to assist in heating the oil in said still. If desired,

branches from said pipe 30 may lead to the other stills of the series.

The operation of the apparatus will be readily understood. The oil, as is well known, frequently contains more or less earthy matter and a small percentage of water, and the first or preparatory still is therefore equipped with the heating coil, as described, to the end that the oil charged into said still may, as it passes about the pipes of the coil, be acted upon throughout its body by the heat radiating therefrom, so that the water will be thoroughly separated from the oil and the earthy matter precipitated. Any suitable cock-controlled outlet 29 is provided for drawing off the water and the precipitated earth from the bottom of the preparatory still. Incidental to the treatment of the oil in the first still, vapor will be formed, which will, of course, pass off through the vapor pipe 16. The oil itself, freed from water and earthy matter, passes continuously from the preparatory still into still No. 2, wherein it is heated and the lighter ingredients vaporized, the vapor passing out through the vapor pipe 16 of said still No. 2. From still No. 2 the oil continuously passes through the short connection to still No. 3, wherein it is subjected to a suitable degree of heat and certain of its constituents vaporized, the portion not vaporized at the temperature maintained in said still No. 3 passing through the short connection to still No. 4, and so on. The height of the receiving mouths or openings of the regulating pipes 21 of the connections 19, through which the oil from the individual stills passes into such connections and the succeeding stills, controls, of course, the liquid level in the several stills. Therefore, by providing the elbows 20 at the receiving sides of the connections 19 with regulating sections 21 of suitable length, the liquid level may be maintained within the several stills at any desired graduated elevations. This feature is of importance. By arranging said liquid receiving mouths of the several connections at successively lower levels in the series of stills, the liquid will be caused to flow by gravity through the series without requiring the aid of a pump or other artificial assistance. The feature of preserving a graduated liquid level in the stills is, furthermore, of importance with respect to those stills which receive the heavier oils after the lighter oils have been vaporized. When, for example, the oil has passed through the first few stills of the series, and approximately 50 per cent.

of it has been vaporized, the oil remaining requires special operations to secure the best results.

It is desirable that the distillation shall be carried on in such manner as will occasion the production of a satisfactory quality of distillates and at the same time avoid impairing the quality of the residuum. Accordingly, when the oil has been reduced by the vaporization of approximately half its mass, it is desirable in operating upon it in the remaining stills that the cubic space above the surface of the liquid in the stills should be very large and that the wall or walls of said still or stills should be remote from the direct action of the fire.

By reason of the high heat to which the oil is subjected, the vapors produced in a given still in the vicinity of the latter end of the series may include not only the particular vapors which it is desired to produce by the vaporization of the oils in said still, but also a certain amount of the heavier products or constituents which it is desirable to have retained in the residuum; and said heavier products or constituents would, in the absence of special provision to the contrary, pass out through the vapor pipe from said still, with the result that the vapor so passing out would not be of required quality, and the mass of residuum, which is received in and is pumped from the last still of the series for further operations in other apparatus, would be deprived of valuable characteristics Accordingly, in operating upon such oil, Livingston prefers to maintain only such quantity of it in a still as will fill it about one-third full, so that large vapor spaces above the liquid levels exist in the last stills of the series. The vapors formed rise in such vapor spaces and circulate through them and about them and in contact with the upper walls of the stills, which are, as stated, out of contact with the fire, with the result that such of the heavier products naturally belonging in the residuum as are carried up into the vapor spaces referred to, will, in circulating about therein, becoming slightly cooler, fall back into the liquid and be transported to the next still and so on.

The operation of distilling is designed to be carried on in this apparatus until a heavy residuum remains, and this residuum is pumped from the last still or last several stills and conveyed away for special operations—among which are included operations for separating it into sundry valuable products.

When, after continued use of the apparatus, one or two of the stills have accumulated a deposit of coke and it is desired to clean

them, this may be done without interruption of the operation of the apparatus as a whole, as will be readily understood. If, for example, it is desired to clean out stills Nos. 6 and 7, the cocks in the two short connections 19 between stills 5 and 6 and 7 and 8, respectively, are to be closed; thereupon said stills 6 and 7 are, so to speak, cut out and isolated. The cocks in the long connection 23 are opened. Thereupon, in the operation of the apparatus, the liquid from the still 5, instead of discharging through the connection 19 into still 6, discharges through said long connection 23 past stills 6 and 7 and into still 8, and from still 8 into still 9, and so on. When the connection is established through the connection 23, as will be understood, the stills 6 and 7 are no longer included in the working series, and the oil from said stills may be run off, the fires extinguished, and any desired matter of cleaning or repair attended to. After the two stills cut out of operation for cleaning or repair have been given whatever attention in such connection may be necessary, they are each supplied with a body of oil at an ordinary temperature. The fires are then started beneath them, and the bodies of oil will, of course, be gradually increased in temperature, and the vaporization proceeded with. When the oil in said stills has become heated to a high temperature and the liquid levels therein are approximately the levels at which the oil is ordinarily maintained in said stills, the cocks are opened in the two short connections by which said two stills are connected to the two adjoining stills and the cocks in the long connection are closed.

It will, of course, be understood that the oil is continuously supplied to the preparatory still, and that its flow through the successive stills is ordinarily continuous. The vaporization contemporaneously proceeds in all the stills of the series as the oil flows through them, each still being maintained at that particular heat which experience has demonstrated to be best adapted for producing the particular distillate obtainable from its contents. From the single battery of stills a constant flow of distillate is maintained through all the distillate pipes to the receptacles in which they terminate. The cocks in the short connections are only used when it is desired to cut out of operation two of the stills as referred to, being then employed to cut off such two stills from communication with their neighbors. The arrangement of the connections is such that those connections 19, which occur at the front of the apparatus, are the

connections which unite among others the individual members of the respective pairs of stills which are at times, as explained, cut out of operation by pairs. As there is no necessity for cutting off connection between the two members of such a pair, the connections which unite the members of such respective pairs are not provided with cocks and are placed at the front of the battery of stills.

The advantage of this arrangement is that all the cocks in the short connections are arranged at the rear of the stills, where the heat is considerably less than at the front.

As explained, the heating mechanism or fire chamber of each still is controllable independently of, and is unaffected by, those of the other stills, and, as a result of this arrangement, the different stills may be maintained constantly each at its own temperature such as will insure the best results. The arrangement of the fire chambers, regulating sections 21, and stills is such that the fire and products of combustion cannot come into contact with the walls of the stills above the level of the liquid therein. This is especially important where oil of the grade termed 50 per cent. oil and oils below such percentage are being operated upon, inasmuch as it enables the still walls above the liquid level to remain at a comparatively low temperature, to occasion the cooling off of the vapor and to promote decomposition.

In the form of apparatus shown in Fig. 185 is shown a modified arrangement of short connections. The short connections therein referred to are designated 32, and these correspond in general location and arrangement with the short connections 19, shown in plan view in Fig. 182, connecting successive pairs of the stills at alternately opposite ends thereof. Each short connection 32 is at its respective extremities provided with branches which lead into the interiors of the respective stills connected. The receiving branch of each such connection is at its inner end provided with a vertically arranged feeder pipe 33, the upper end of which extends to a point above the desired liquid level in the still in which it is situated, and the other to a point in the vicinity of the bottom of said still. Both ends of feeder pipes 33 are open, and the bodies of these pipes are in direct communication with the interiors of the receiving branches of the short connections 32. In this construction the oil is taken from the bottom of the several stills to supply the succeeding

stills. The preferred form of short connection, however, is that shown with the two right-hand stills of Fig. 183, in which the receiving regulator pipe 21 of each is equipped with a sleeve 35, encircling it and making a liquid-tight connection with the receiving branch. This sleeve, which acts as a feed controller, is open as to both ends, one end extending into the vapor space above the liquid level, and the other to a point near the bottom of the still.

In this arrangement, while the liquid level inside the sleeve will be the same as that outside, all liquid within the sleeve will gain access thereto through its lower end, and all liquid drawn from the still through the pipe 21 will thus be taken from the bottom of the still. The mouth of the pipe 21 constitutes the outlet from the still and controls the liquid level therein to the extent that the liquid is not in the ordinary operation of the apparatus drawn down to a level below that of said mouth.

**Other Processes of Continuous Distillation.**[1]—While, as indicated above, the continuous distillation of petroleum received its real industrial impetus from the successful operation of the apparatus invented by Livingston, this system has attracted attention in the United States since 1860, in which year a patent was granted to D. S. Stombs and Julius Brace for certain improvements in the distillation of coal oil (Fig. 186). This invention was described as "an improved method or process for separating the various products contained in crude coal oil or other oils by a continuous successive distillation, in such manner that, on the operation being once commenced and a fresh supply of crude oil regularly maintained, a simultaneous discharge of several qualities of oils and of other products, by a simple process, may be continued for an indefinite period of time without any interruption, and without any care or trouble of removing the products from one receptacle to another, or of clearing the distilling vessel of the residuum, the same being performed by regular transference of the unfinished material from one retort to another, and the final discharge of the last waste product from the last retort in the apparatus without any mechanical aid whatever, the fluid discharging itself by the regular working of the apparatus."

[1] No attempt is made to include accounts of all systems and processes proposed, but rather to point out, historically, the course of invention in this field, both in the United States and in Europe.

In the use of the apparatus described in their specification, Stombs and Brace direct that crude oil is to be delivered into still *A*, through the pipe *A*, until the level of the oil reaches the point of discharge of the pipe *D* into the retort *B*. The still *A* is then heated to 170° to 200° (presumably degrees C.) so as to vaporize the more volatile products, which pass through the pipe *H* to the condenser. When this volatilization has been effected, a further quantity of crude oil is slowly introduced through the pipe *A*. The crude oil already in the still having had its specific gravity raised by the elimination of it-

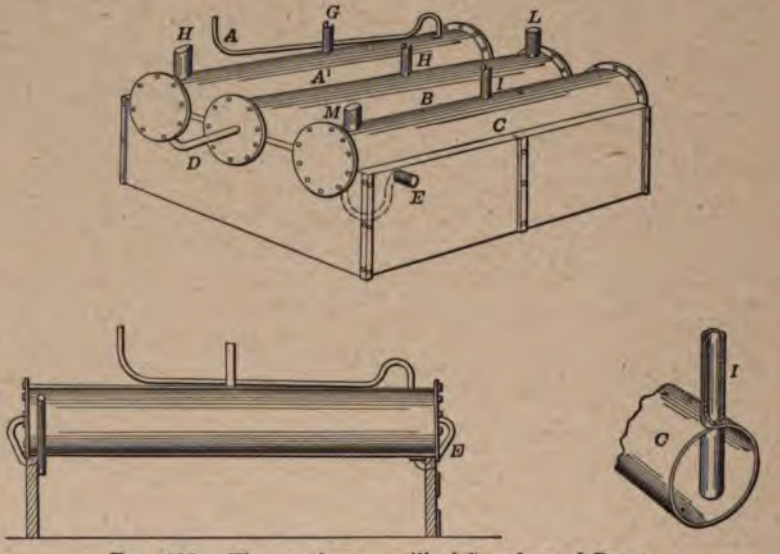



FIG. 186.—The continuous still of Stombs and Brace.

more volatile constituents, the fresh oil will float upon its surface, and will there be subjected to a process of fractional distillation. As the level of the oil in still *A* becomes raised by the introduction of fresh crude oil, the heavier oil will begin to flow into still *B*, through the pipe *D*. Still *B* should then be heated to about 300° (presumably degrees C.), and from it a product of higher boiling point than that obtained from still *A* will pass away, through the pipe *L*, to the condenser. When still *B* has thus become filled so that a portion of its contents is flowing into still *C*, the latter should be heated to incipient redness, and products of higher boiling point than those yielded by still *B* will, in like manner, pass through the pipe *M* to the condenser.

When still $C$ has become filled to a sufficient height, the discharge of the residuum through the pipe $F$ will commence, and the whole process will thereafter be continuous, as long as the supply of fresh crude oil to still $A$ is maintained and the stills are kept at the requisite temperatures.

The patent of A. H. Tait and J. W. Avis, granted in 1867, pertained to "improvements in apparatus for distilling petroleum, etc." (see Fig. 187). In the specification of Tait and Avis the apparatus was described as consisting "mainly of a still divided by one or more partitions into two or more compartments, which communicate with each other at the top by suitable openings for the vapors, and at the bottom by openings for the liquid." The openings at the top are provided with gates or cocks "to shut off the communication and divide the light from the heavy vapors, so as to pass each oil into its appropriate condenser."

"The compartments next to the fire are also provided with gates or valves to the openings at the bottom, so that, on closing these, the compartment may be cleaned out without emptying the entire still. The crude oil is admitted to the compartment farthest from the fire, and the compartment nearest to the fire is connected by a pipe with coking retorts, in which the refuse or heavier parts of the oil are subjected to a final distillation."

$A$ represents the still, divided by partitions, $a$, into compartments, $b$, $b^1$, $b^2$, $b^3$, $b^4$, $b^5$. The crude oil is admitted from the supply-tank, $B$, into the first compartment, $b$, which is farthest from the furnace, and it passes from this compartment to the succeeding compartments through apertures, $c$, in the lower parts of the partitions, $a$, the apertures being so situated that the oil is compelled to run through the still in a zigzag course. In the upper parts of the partitions, $a$, are openings, $d$, for the passage of the vapor, and these openings are fitted with valves, $e$, $e^1$, furnished with handles, $f$, $f^1$. From the compartments, $b^1$ and $b^2$, rise pipes, $g$, $g^1$, provided with stop-cocks, and each of these pipes is connected with one of the condensers, $C$ or $C^1$. If the valve, $e$, is closed, the vapor from the first two compartments, $b$, $b^1$, passes through the condenser, $C$, and the vapor from the other compartments through the condenser, $C^1$. But if the valve, $e$, is opened, and the valve, $e^1$, is closed, the vapor from the compartments, $b$, $b^1$, $b^2$, $b^3$, can be made to pass through either of the condensers by means of stop-cocks. The

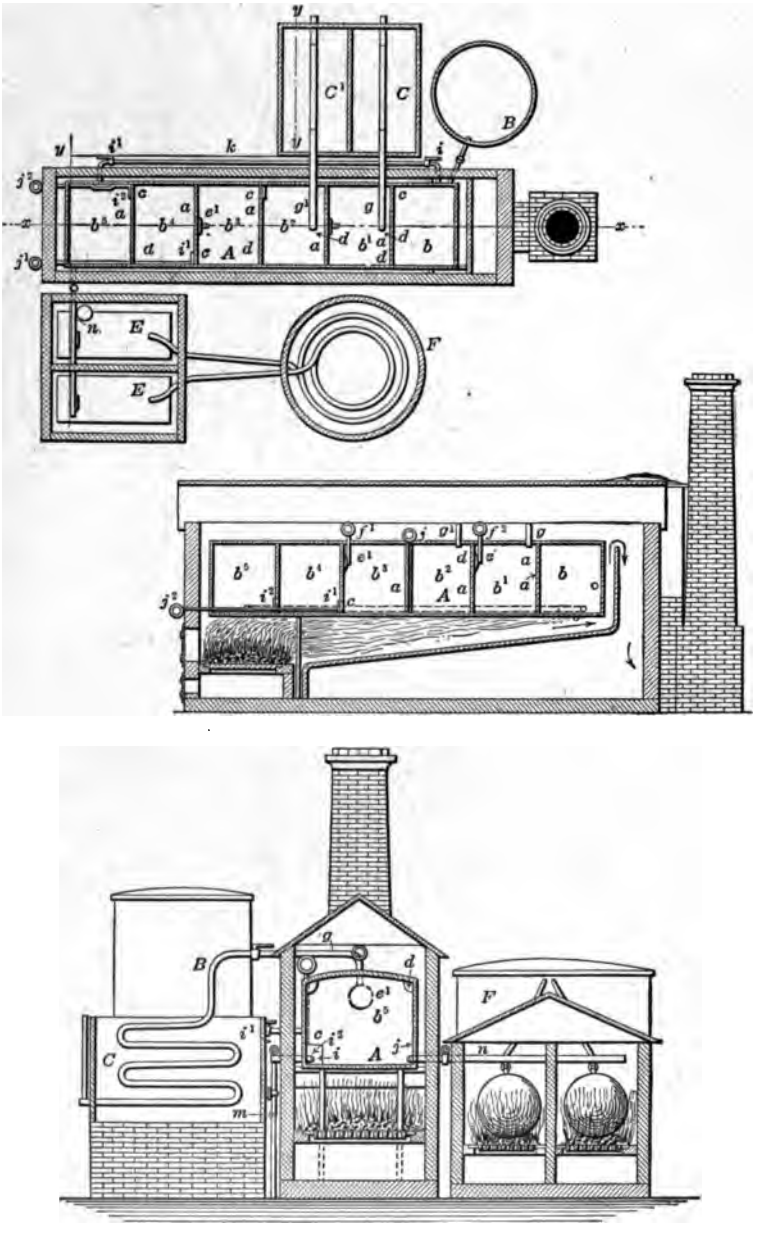



FIG. 187.—The continuous still of Tait and Avis.

apertures, $c$, in the last three partitions, $a$, are provided with gates, $i$, $i^1$, $i^2$, provided with handles, $j$, $j^1$, $j^2$. By closing the gate, $i$, the liquid is prevented from passing into the compartments, $b^3$, $b^4$, $b^5$, which are nearest to the fire, and these compartments can be readily cleaned without interrupting the operation of the remaining compartments. Similarly, by closing the gate, $i^1$, the last two compartments, $b^4$, $b^5$, can be cleaned; and if it is desired to clean the last compartment, $b^5$, only, the gate, $i^2$, is closed, the compartment, $b^5$, being brought into direct communication with the compartment, $b$, by means of a pipe, $k$, situated on the outside of the still. This pipe is provided with stop-cocks, $i$, $i^1$, and is connected with the discharge-pipe, $m$. It is claimed that the liquid from the first compartment can thus be run directly into the last compartment, and the specific gravity of the product obtained from these compartments, as well as from the remaining compartments, can in this way be "regulated and equalized." The compartment, $b^5$, communicates through a pipe, $n$, with the coking stills, $E$, $E$, which are provided with a separate furnace. The vapor from the coking stills passes to the condenser, $F$.

The invention claimed in the patent secured by S. A. Hill and C. F. Thumm in 1870, for "improvements in apparatus for distilling hydrocarbon oils" (see Fig. 188), is described as consisting in "so constructing and arranging a series of stills with relation to each other that the oil will flow over the bottom of the stills in a thin and continuous sheet, with a different degree of heat applied to the bottom of each still; and in so arranging the connection between the several stills that the flow of oil through one or more of the stills may be cut off from the other stills in the same series."

According to the specification of Hill and Thumm, $A$ and $B$ are large separating and heating stills, used for the purpose of separating water from the oil, distilling off the very volatile hydrocarbons, and heating the oil. $A^1$ is a range of furnaces under the stills. $A$ and $B$ are supplied with oil through pipes, $w$, and are furnished with vapor pipes, $x^1$, which may be connected with condensers. Near the bottom of the stills, $A$ and $B$, are two pipes, $h$ and $i$, connected to the pipe, $g$, which communicates with the stills, $C$, $D$, $E$, and $F$, by means of pipes, $k$, $l$, $m$, and $n$. These pipes are provided with stop-cocks, as shown in the illustration. At the opposite end, the stills, $C$ and $D$,

and *E* and *F*, are respectively connected by means of the pipes, *o* and *p*, which are placed close to the bottom. The stills, *C*, *D*, *E*, and *F*, have vapor pipes, *x*, which communicate with condensers. In carrying out the process of distillation with the apparatus, oil is first introduced into the stills *A* and *B*, where the water is separated from it. After the oil has been heated to about 120°F., the valves, 1, 3, and 2, of the pipes, *h*, *g*, and *i*, are opened, and the oil is thus allowed to flow from still, *A*, over the bottom of still, *C*, thence through the pipe, *p*, into still, *F*,

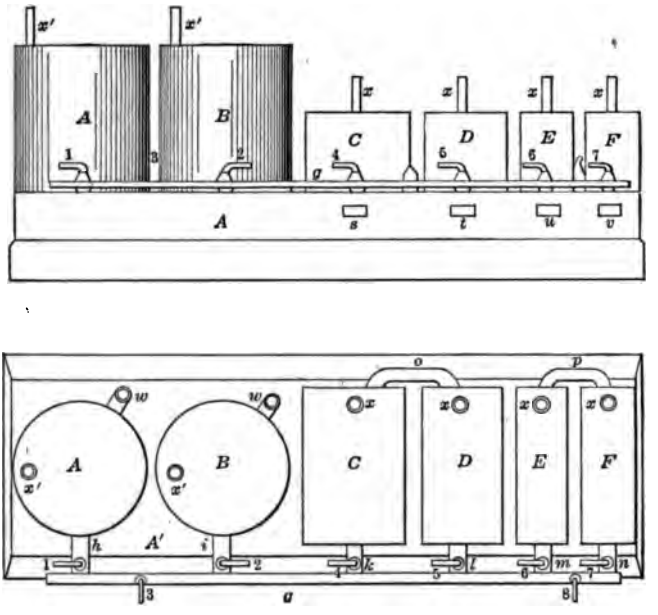



FIG. 188.—The continuous still of Hill and Thumm.

passing over the bottom of each still in a thin and continuous sheet, and being subjected to a gradually increasing heat.

The apparatus of Samuel Van Syckel[1] was operated at one time in a refinery in Buffalo, N. Y. This installation consisted of a series of stills, the supply of oil to which was automatically controlled by means of a float in the feed tank. From the first still naphtha was obtained, while the other two yielded illuminating oil, the residue being removed from the last still.

In the arrangement of James Cole, Jr.,[2] a series of connected

[1] United States Patent 191203, May 22, **1877.**

[2] See CREW'S "Practical Treatise on Petroleum," **1887.**

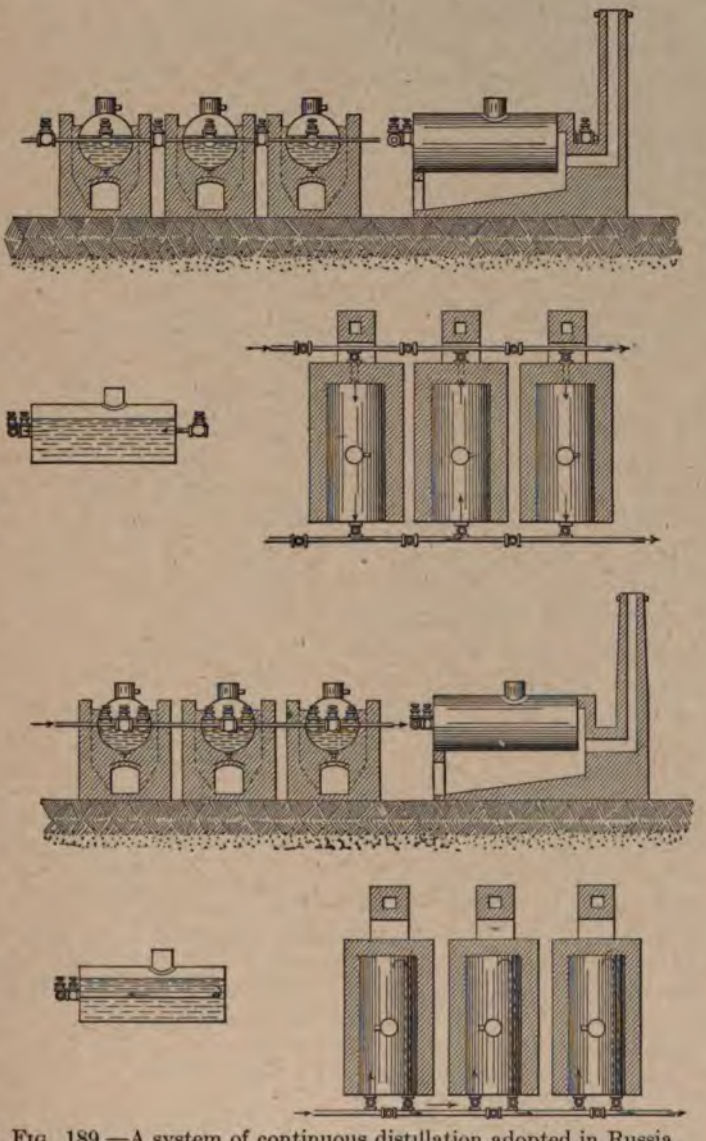


FIG. 189.—A system of continuous distillation adopted in Russia.

stills were maintained at carefully graduated temperatures; the apparatus of Rycroft and Mason[1] consisted of a vertical coiled-pipe retort containing as many chambers as the desired number of fractions, the successive sections being divided by partitions, each a little below the preceding one and arranged in separate spaces of the furnace; and Pielsticker claimed a process wherein the oil, contained in a tank, was heated by a steam coil and was forced by a pump or steam injector into an iron coil, contained in a cylinder filled with metal or salt of known melting point, or embedded in loosely fitting channels made in a block of iron, heating being accomplished in both cases by a furnace.[2]

The system of continuous distillation was adopted by Nobel Brothers, owners of one of the most important of the Baku refineries, in the production of illuminating oil in 1883, and was later used by the same firm in the manufacture of lubricating oils. According to the system employed (see Fig. 189), the oil flows through a series of fourteen or sixteen stills heated to successively higher temperatures; before entering the first of these stills, the crude oil passes through a "fore-warming" still, in which the temperature is raised to 200°C., and, in its passage through the stills, the oil is further heated gradually, leaving the last still at a temperature of 300° to 320°C.[3] Another continuous distillation apparatus which has been used in Russia is that of Schukov, Intchik and Bary;[4] this consists of a metallic vessel fixed above an ordinary still and divided by a number of flat pans into a series of compartments in which constant temperatures are maintained. The apparatus of Alexieev,[5] in which the superheated steam ordinarily used is replaced by light hydrocarbons, the more volatile products obtained in the distillation of petroleum being separately collected and pumped back into the still, has not, according to Veith,[6] worked successfully in the Baku

[1] See United States Patent 339545 of **1886**, and English Patent 249 of **1891**.

[2] English Patent 6466, Apr. 28, **1890**.

[3] On the NOBEL distilling battery, see GADASKIN and POPICH, *J. Russ. Phys.-Chem. Soc.*, **44**, 1715.

[4] VEITH'S "Das Erdöl," **1892**; *Mem. Imp. Russ. Tech. Soc.*, May, **1889**.

[5] On this system, see MEIRO, *Zap. Imp. Russk. Tekhn. Obsch.*, **24** (1890), December, p. 1.

[6] *Loc. cit.* Cf. French Patent 379521, July 3, **1907**, of WELLS, wherein an absorbent gas is used to carry over the vapors of the oils in the order of their boiling points.

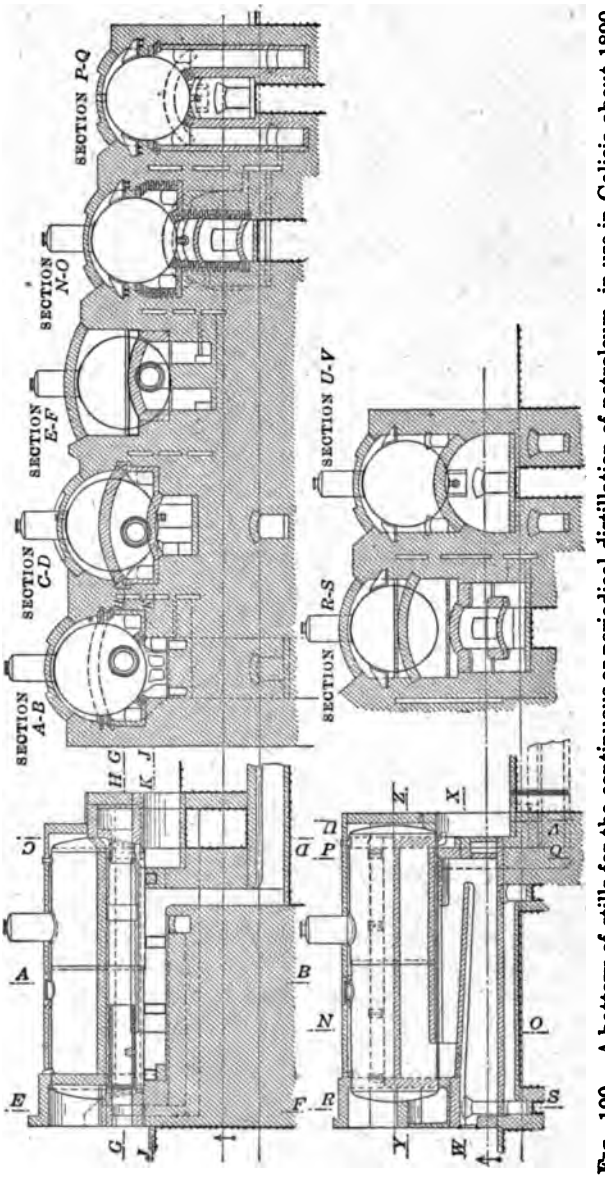



FIG. 190.—A battery of stills for the continuous or periodical distillation of petroleum, in use in Galicia about 1890. (After Boverton Redwood.)

refineries in which it has been tried. Fig. 190 shows a battery of illuminating oil stills in use in Galicia about 1890.

About 1900 it was recognized among many European refiners that the continuous system possessed definite advantages over the intermittent system of distillation. For instance, there was the saving of fuel, through the non-necessity for cooling down and reheating the stills, and owing to the manner of setting the stills in brickwork; moreover, the waste heat was employed for preliminary heating. It has been ascertained that an equable temperature could be ensured by having a large heating surface and maintaining the oils at a constant level.

However, an objection to continuous distillation, based on the adverse experience gained in Rumania with petroleum containing benzine, was that the benzine could not be so effectually removed at the beginning of the process as to give products of equal flashing-point with those furnished by intermittent stills.[1] Von Grölling claimed that this was obviated by the Grölling-Estlander system, in which the heat of the stills is utilized for preliminary heating and predistilling, and the still is connected with a plate dephlegmating column of special construction, dephlegmating condenser, and final condensers.[2] A water injector controls and reduces the pressure at the top of the plate column, and, by its water spray condensing the volatile gases, is said to prevent considerable loss; a vessel for the separation of the condensed vapor and the water is provided, and the liquid of lower boiling point is removed from the base of the plate column, direct steam or a steam jacket being used at this point of heating.

The process of E. A. Barbet[3] for the continuous rectification of petroleum and volatile hydrocarbons, is of more than passing interest. The crude oil is passed through a series of heaters and column stills, fitted with condensers and means for collecting and reboiling the various fractions; the light oils are eliminated first, then the illuminating oils, and finally the heavy oils are cracked and the fractions are separated in a column apparatus. Barbet has also devised a simplified apparatus for separating, by a continuous process, four fractions boiling below 130°C. and an

[1] *Petrol. Ind. Techn. Rev.*, **2**, 130.

[2] See French Patent 322789, July 4, **1902**; and English Patent 29705, Dec. 21, **1910.**

[3] French Patent 322265, June 7, **1902.**

illuminating oil, and for cracking the heavy oil not distilled;[1] it comprises a tubular heater with an inclined grate for decomposing the heavy oils, a large boiler communicating with a fractionating column, and condensers and coolers for the products.[2] A later patent of Soc. E. Barbet et Fils et Cie[3] relates to the continuous fractional distillation of petroleum by passage through a series of bulk evaporators, maintained at successively increasing temperatures and under the same pressure. The apparatus is so arranged that the vapors generated in an evaporator working at a higher temperature are employed for heating the evaporator at the next lower temperature, the vapors being made to circulate below the evaporator heated by them in the opposite direction to that taken by the liquid circulating in that evaporator.

Other patented processes of continuous distillation are the following: That of Josef Fischer,[4] in which the various constituents are passed horizontally and independently between heating partitions; the process of Eugen Wernecke,[5] wherein direct-fired cast-iron apparatus comprises an obconic exteriorly heated distillation chamber, provided on its inner walls with cups formed of replaceable rings decreasing in size from the top to the bottom and having taps from which the liquids of different specific gravity may be drawn off; and the apparatus of Crolbois,[6] which consists of superposed hollow vessels, externally heated and so arranged that the liquid is caused to flow in a film over the inner surface of the upper part of the vessel before arriving at the lower part.

The specifications for a continuous "topping" plant have been published by Prutzman in the *California Derrick*, **3**, Nos. 11 and 12.

[1] First addition, dated Dec. 1, **1906**, to French Patent 322265, June 7, **1902**.

[2] The crude oil is admitted to the fractionating column some distance above the base, and three fractions are drawn off from the upper part of the column. The lightest product is obtained from the vapor not condensed in the column. Illuminating oil is drawn off below the inlet for crude oil or from the boiler itself, and the non-volatile residue passes to the tubular heater.

[3] English Patent 9088, Apr. 9, **1914**.

[4] German Patent 184226, June 21, **1905**.

[5] German Patent 201372, Apr. 28, **1907**.

[6] English Patent 7391, Mar. 24, **1911**.

## THE "CRACKING" PROCESS

The so-called "cracking" process, which was noticed by Silliman in 1855 and was again "accidentally discovered" in 1861,[1] may be said to consist in distilling petroleum or its products at a temperature higher than the normal boiling points of the constituents which it is desired to decompose. Such a result may be effected by distillation under pressure or

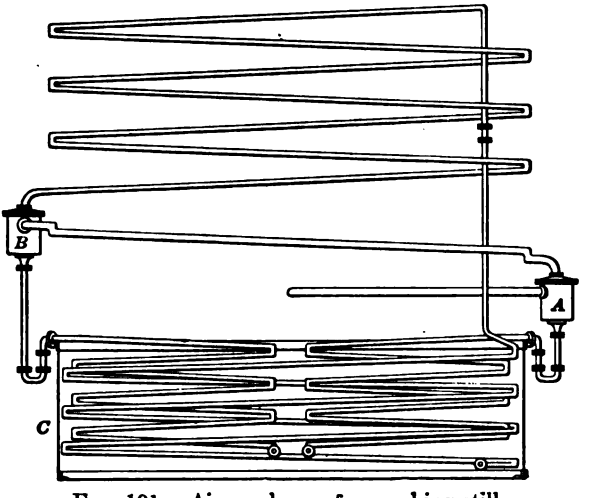



FIG. 191.—Air condenser for cracking still.

by allowing the condensed distillate to come into contact with a highly heated surface, by which means comparatively heavy

[1] See PECKHAM, *Am. J. Sci.*, (2), **47** (1869), 9; SILLIMAN, *Am. Chemist*, **2** (1871), 18 and 20; LEET'S "Petroleum Distillation," New York, **1884**; and AISINMANN'S "Die Destruktive Destillation," p. 3. According to LEET, a workman in charge of a 16-bbl. still at Newark, N. J., built a "strong fire" under the still after about half its contents had been distilled off and left with the intention of returning within an hour. It was not, however, until after 4 hr. that he returned to the refinery, and he then found that a light-colored distillate, of lower gravity than that which was being obtained when he left, was issuing from the condenser. As the result of experiments to which this discovery led, it was found that a portion of the distillate had condensed upon the cooler upper part of the still, and had dropped back into the still, where the temperature was sufficient to cause its dissociation into products of lower boiling point. These and other similar observations soon led to the setting of stills with the upper parts wholly uncovered, so that the desired partial condensation could occur and thus convert the less valuable intermediate hydrocarbons into kerosene in the manner described.

oils may be made to undergo dissociation into specifically lighter hydrocarbons possessing lower boiling points.

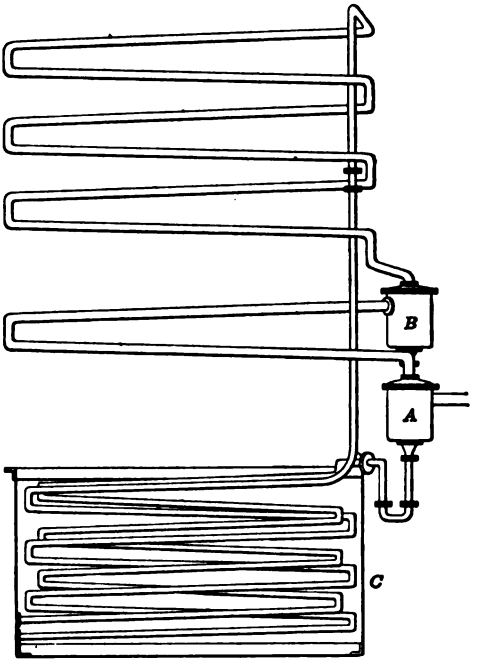



FIG. 192.—Air condenser for cracking still.

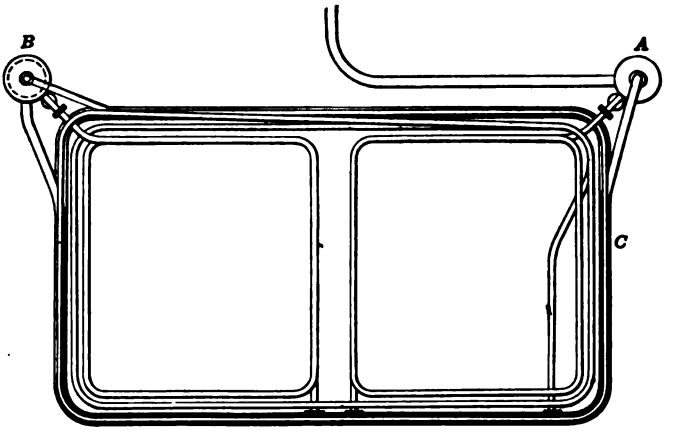



FIG. 193.—Air condenser for cracking still.

It is almost impossible to decompose heavy petroleum oils without the production of more or less low-boiling hydrocarbons,

no matter what method is employed, provided a suitable temperature—say 380° to 450°C.—prevails somewhere in the series of operations. The principal difficulty in large scale operations is the deposition of coke.

The earlier attempts to effect the conversion of heavy petroleum hydrocarbons of high boiling points into lighter, more volatile products were conducted in most, if not all, cases with the object of increasing the yield of illuminating oil. In fact, the "cracking" process has been extensively employed in petroleum refineries for converting intermediate hydrocarbons into kerosene,[1] and a large number of arrangements for carrying out the process have been proposed and patented.[2]

A cylindrical cracking still having a total capacity of about 200 bbl., has been used in certain American refineries. Figs. 191, 192, and 193 show forms of an air condenser combined with a water-cooler which have been employed in connection with such stills. The vapors pass from *A* to *B* and thence through the water-cooler *C*.

A cast-iron still of the type used in Russia in conducting the cracking process, is shown in Fig. 194. It is in the form of a cast-iron cylinder with a hemispherical bottom, having an outlet provided with a cock, through which the residue may be removed.

[1] It has been stated (see VEITH'S "Das Erdöl," **1892**) that, when cracking is conducted in uncovered stills (see preceding footnote), the product is not identical with, and probably not equal in quality to, the natural distillate. It is asserted that such oil is inferior in light-giving power and more readily acquires a dark color on keeping. It is said that the odor of the cracked oil is stronger, and that in the chemical treatment a much larger quantity of sulphuric acid is required. The cracking does not commence until after the more volatile products, including the distillate from which high-test oils having a fire test of from 120° to 150°F. are made, have been obtained. The fires are then damped, and the distillation continued slowly, to permit of the necessary condensation. During the cracking, a considerable quantity of permanent gas is evolved, and this is usually burned beneath the still. See pp. 455 and 724.

[2] The earliest patent directly bearing on the subject is English Patent 3345 of 1865, issued to JAMES YOUNG for certain "improvements in treating hydrocarbon oils." In this patent, the distillation is described as being conducted in a vessel having a loaded valve or a partially closed stop-cock, through which the confined vapor escapes under any desired pressure. Under such conditions, distillation takes place at higher temperatures than the normal boiling points of the constituent hydrocarbons of the oil, and a partial cracking results. The patent was taken out for the treatment of shale oil, a pressure of about 20 lb. to the square inch being recommended.

In some cases distillation is carried to dryness, the coke being then removed through a manhole. When superheated steam is employed in the distillation, as is sometimes the case, the coke is of a somewhat soft nature and consequently does not adhere firmly to the still. The crude oil, or residue from a previous distillation, is placed in the still and heated, at first gently, to volatilize any water present and thus prevent "priming," or "bumping," and then to a higher temperature to distil

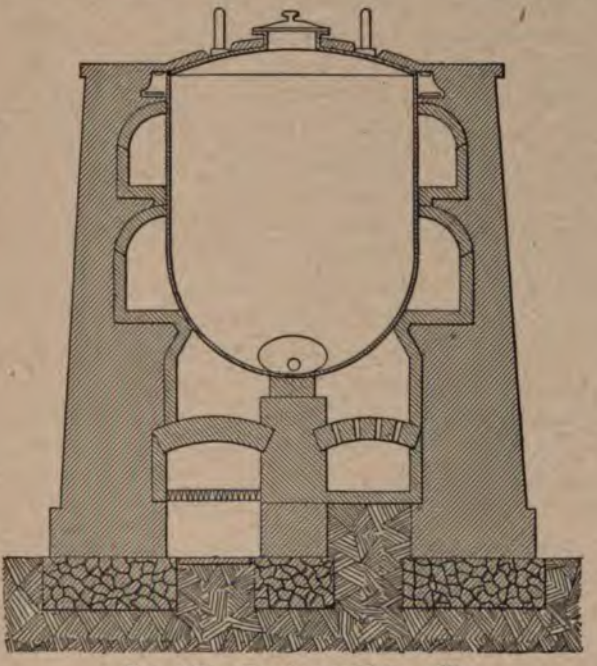


FIG. 194.—Russian cracking still.

off the more volatile oils. Finally, the cracking process commences, the heating being regulated by the dampers according as lighter or heavier distillates are required.[1] A suitable dephlegmator is used to collect and return or remove the heavy oils which are carried over during the distillation.

[1] To save room and time in the preliminary heating, it is usual, when the distillation is nearly completed, to refill the still without removing the residue; but it is said that this should not be done too often, or the coke-incrustation may become excessive, the distillation being thus retarded and the still injured by overheating. The dimensions of the still vary considerably, but the height is generally two or two and a half times the diameter.

The preparation of gasoline and kerosene from heavier hydrocarbons has been considered at some length by Brooks, Bacon, Padgett and Humphrey,[1] from whose report much of what follows has been taken.

**The Production of Gasoline and Kerosene by "Cracking."**—The "cracking" of heavy hydrocarbons by heat is to be regarded as simply an instance of the general rule that organic compounds are decomposed by heat. It is well known that the simpler petroleum hydrocarbons are stable at much higher temperatures than those of higher molecular weight. Every refiner knows that certain crudes "crack" more easily than others, which fact is to be accounted for by the presence of hydrocarbons of different constitution.[2] In the case of the simpler hydrocarbons which have been studied, it is known that olefines are in general less stable to heat than saturated hydrocarbons of the same molecular weight, and naphthenes are usually more stable than paraffins. Normal hexane and the more stable methyl cyclopentane are good examples of the latter types. It was to be expected, therefore, that attempts would be made to produce gasoline from heavier hydrocarbons by heat decomposition at temperatures somewhat higher than those employed for the production of kerosene by cracking. A large number of recent patents seek to attain this end.[3] The fatal difficulty with processes of this class, operating at atmospheric pressure, is the large per cent. of olefines contained in the resulting gasoline. This figure will vary, when the oil is simply subjected to heat at atmospheric pressure, from 20 to 50 per

[1] *J. Ind. Eng. Chem.*, **7** (1915), 180. For a detailed resume of the cracking and distillation of petroleum under pressure, see Brooks, *J. Frank. Inst.*, Dec., **1915**, 653–72; and on the pyrogenesis of hydrocarbons, see *J. Ind. Eng. Chem.*, **9** (1917), 879–902. On gasoline from synthetic crude oil, see Snelling, *Bull. Am. Inst. Min. Eng.*, **1915**, No. **100**, 695.

[2] On this point, see Zaloziecki, *Z. angew. Chem.*, **1897**, 619. On the stability of paraffin hydrocarbons, see Egloff and Moore, *Met. Chem. Eng.*, **16** (1917), 47.

[3] On increasing the available supply of gasoline by cracking and for a statistical review of the question of gasoline supply, see Brooks, *J. Ind. Eng. Chem.*, **7** (1915), 176. On the production, composition, properties and uses of petrol and its substitutes, see Lewes, *J. Roy. Soc. Arts*, **63** (1915), 757, 773 and 792. On gasoline, see also p. 870; and on petrol, see p. 897. On the history of cracking and the Hall cracking process, see Chandler, *Met. Chem. Eng.*, **13** (1915), 779; *J. Ind. Eng. Chem.*, **8** (1916), 73. On the general methods of cracking petroleum oils, see Ellis and Wells, *J. Ind. Eng. Chem.*, **7** (1915), 1029. For a select bibliography of the pyrogenic reactions of hydrocarbons, see Rittman and Whitaker, *Technical Paper* **120**, *Bureau of Mines*, **1915**, 14–15.

cent., depending on the temperature at which the oil is cracked. The maximum per cent. of olefines demanded by the simple general equation, $RCH_2 - CH_2 - CH_2R_1 \rightarrow RCH_3 + CH_2 = CH.R_1$, is seldom attained owing to other reactions, such as polymerization of the olefines and the formation of naphthenes. Gasoline made by cracking under atmospheric pressure the heavier portion of Oklahoma crude, *i.e.*, the part left after distilling off the gasoline and kerosene, in such a way as to condense and return to the still all but gasoline and naphtha, showed a content of olefines of approximately 28 per cent., as indicated by the iodine number and loss to ordinary sulphuric acid. Consequently a number of patents have been issued covering processes which seek to hydrogenate the olefines formed. It has been proposed[1] to hydrogenate the olefines by placing a catalyst, such as platinum or palladium, in the still, but this process is not being operated for obvious reasons. Several proposed processes claim the cracking of heavy hydrocarbons in the presence of steam and iron. The method patented by the New Oil Refining Process Company, Ltd.,[2] requires the passing of petroleum and steam through a retort containing scrap iron at 540° to 650°C. The processes proposed by Adams,[3] Greenstreet,[4] Hyndman,[5] and Turner[6] specify the cracking of heavy oil at higher temperatures (Greenstreet and Hyndman specify a "cherry temperature") in the presence of steam alone. The claim has been made that the water is converted into carbon monoxide and hydrogen, the latter effecting hydrogenation of the olefines produced. Testelin and Renard[7] vary this by passing the vapors of oil and steam over a red-hot layer of clay or alumina. Vernon Boys[8] passes the oil and steam mixture through heated tubes containing nickel rods. Lamplough[9] cracks in the presence of steam and in contact with nickel in a retort maintained at a dull red heat. Moeller and Woltereck[10]

[1] United States Patent 826089, July 17, **1906.**

[2] French Patent 451471, Dec., **1912**; English Patent 28460, **1911**; English Patents 20074 and 20075, Sept. 3, **1912**; English Patent 13675, **1908.**

[3] *Petrol. Gaz.*, **1910**, No. 7, 2.

[4] United States Patent 1110925; English Patent 16452, July 13, **1912**

[5] French Patent 462484, Sept. 11, **1913.**

[6] French Patent 451162, **1912.**

[7] German Patent 268176, Oct. 12, **1913.**

[8] *Met. Chem. Eng.*, **1914**, 180.

[9] English Patent 19702, Aug. 28, **1912.**

[10] English Patent 16611, July, **1913.**

pass oil and steam through tubes containing coke at 600° to 700°C. Sabatier and Mailhe[1] convert crude petroleum, etc., into hydrocarbons distilling below 300°F. by passing the vapors over catalyzing metals in a state of division in contact with metals or alloys of metals of the nickel series maintained at a temperature varying from 300°F. to bright red. None of the above processes is being operated on a large scale in this country at the present time.

Another method of subjecting the heavy petroleum hydrocarbons to the higher temperatures required to produce the desired increased yields of gasoline is heating or distilling under pressure. It is known that unsaturated hydrocarbons are condensed to saturated hydrocarbons by the action of heat and pressure. Ipatiew[2] showed that ethylene, probably the most stable of the olefines, could be converted into a mixture of liquid hydrocarbons at 325°C. and 70 atmospheres pressure. At 380° to 400°C. the reaction was sufficiently rapid to cause a fall of 5 atmospheres per minute in the pressure of the closed apparatus. Engler and his collaborators have shown[3] that amylene and hexylene yield saturated compounds of the naphthene type by heating under pressure. It is therefore to be expected that naphthenes will be found in gasoline and kerosene made by pressure distillation of heavier hydrocarbons; indeed, this has been shown by Engler.[4]

In regard to the pressure distillation processes which have been proposed, the older ones sought to increase the yield of kerosene, since gasoline, in the days of such early patents as those of Benton, was of very little value. In the present discussion, only the more important processes are mentioned. In 1869, Peckham[5] claimed to have obtained, by distilling under 20 lb. pressure, as much as 60 per cent. *illuminating oil* of specific gravity 0.810 from a California crude oil yielding 20 per cent. kerosene of the same quality when distilled in the usual manner at atmospheric pressure. Thorpe and Young[6] obtained from paraffin, by heating at a "high pressure" and subsequent

[1] United States Patent 1124333, Jan. 12, **1915.**

[2] *Ber.*, **44** (1911), 2978.

[3] *Ibid.*, **42** (1909), 4610, 4613, 4620; **43** (1910), 388.

[4] ENGLER, *ibid*, **33** (1900), 2915; NASTYUKOV, *J. Russ. Phys.-Chem. Soc.*, **36** (1904), 881; KRÄMER and SPILKER, *Ber.*, **33** (1900), 2265.

[5] *Chem. News*, **1869**, 183; *Am. J. Sci.*, (2), **47**, 9.

[6] *Proc. Roy. Soc.*, **19** (1871), 370; **20** (1872), 488; **21** (1873), **184.**

fractionation, a light oil shown to contain pentane, hexane, heptane, octane and nonane.

A few patented processes,[1] including the recent ones of Testelin and Renard,[2] subject oil to heat and pressure and distil the liquid *after* the pressure is released. Testelin and Renard seek to avoid the deposition of carbon, always obtained when oil or oil vapor is passed through a heated pipe coil, as in some of the earlier processes. This they propose to do by uniformly heating the pipe coil, containing the *liquid oil*, to a temperature not exceeding 450°C., by submerging the coil in a bath of lead at the desired temperature. A sufficient pressure is maintained on the heated oil to keep it in a liquid state. The process patented by Clark[3] describes pumping the oil through heated

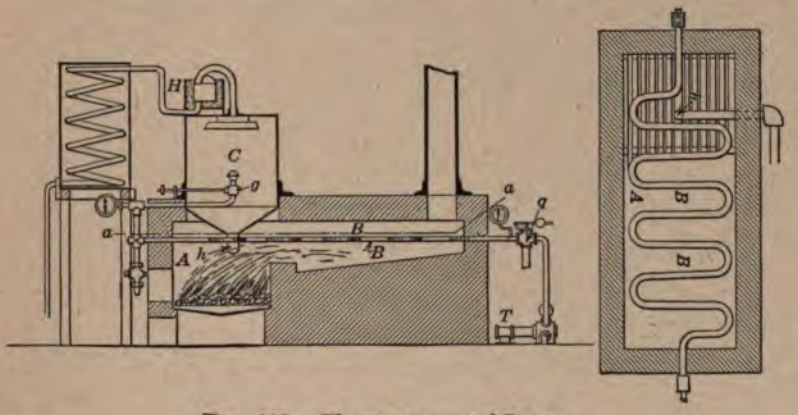



FIG. 195.—The apparatus of Benton

pipes, collecting the hot oil in a receiving drum where distillation under pressure is permitted. The older patents of G. L. Benton[4] also embodied the principle of subsequent distillation after release of the pressure, but heated the pipe coils directly by a flame. The process of Benton consisted in pumping "the refuse of petroleum oil, tar oils, and other heavy mineral oils," under a pressure of 285 lb. or more to the square inch, through a series of pipes heated in a furnace, and allowing the products

[1] SCHUKOV and GAVRILOV, *Z. angew. Chem.*, **1893**, 231.

[2] RENARD, English Patent 3413, Feb. 10, **1913**.

[3] United States Patent 1119496, Dec. 1, **1914**.

[4] United States Patent 342564 and 342565, May 25, **1886**. On BENTON'S apparatus, see *J. Ind. Eng. Chem.*, **7** (1915), 573.

to escape into a vapor chamber. The apparatus, which was said to produce lighter hydrocarbons (kerosene) from the products specified, is illustrated in Fig. 195. It consists of a series of pipes, *B*, heated by means of a furnace, *A*, and communicating on one side with a force-pump, *T*, and on the other with a vapor

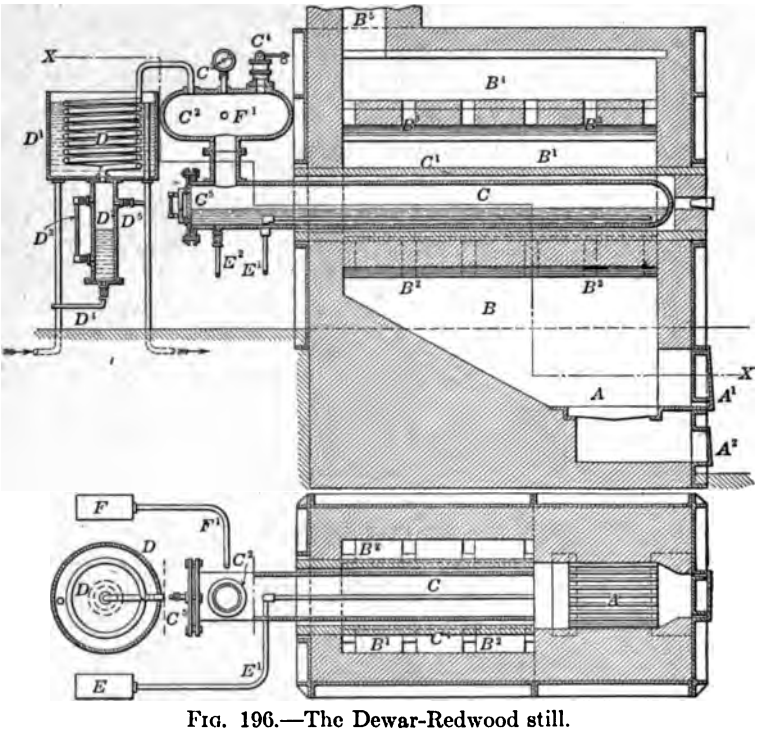



FIG. 196.—The Dewar-Redwood still.

The lower portion of the figure is a sectional plan on the line *XX*. *A* is a fireplace, with fire-door, $A^1$, and ashpit door, $A^2$, suited for regulating the admission of air as required. Instead of a fireplace with grate for burning solid fuel, it is said that a burner for solid or gaseous fuel may be employed. Above the combustion-chamber, *B*, is placed a metal retort, *C*, which is enclosed within a refractory casing, $C^1$, to protect the metal from excessive local heating. The encased retort is situated in a heating-chamber, $B^1$, into which the hot products of combustion ascend by side ports, $B^2$, and from which they pass by central ports, $B^3$, into a flue, $B^4$, communicating with a chimney, $B^5$. The front part of the retort *C* communicates freely with a still-head, $C^2$, provided with a pressure-gauge, $C^3$, and safety-valve, $C^4$ The exposed end of the retort *C* is closed by a readily removable cover, $C^5$, provided with a glass gauge to show the level of the liquid in the retort. *D* is a pipe-coil situated in a tank, $D^1$, in which circulation of water is maintained. The upper end of the coil *D* communicates by a pipe with the still-head $C^2$, and its lower end opens into a hollow column, $D^2$, which is provided with a glass gauge, $D^3$, and has at the bottom an outlet-pipe, $D^4$, furnished with a stopcock or valve. *E* is a pump for forcing the oil to be treated, by a pipe, $E^1$, into the retort *C*, this pipe preferably extending to the further end of the retort. By another pipe, $E^2$, furnished with a cock or valve, the contents of the retort can be drawn off, or this pipe may communicate with a second retort, which in like manner may be connected with a third, so as to form a series of any required number. *F* is an air-compressing pump, by which air or suitable gas is forced by a pipe, $F^1$, into the still-head $C^2$, or it might be into any other part of the apparatus which is in communication with the still-head. The retort *C* being partly charged with oil by the pump *E*, and the spaces in the retort *C*, in the still-head $C^2$, and in the condensing-coil, *D*, and the column $D^2$, being charged with air or gas at the desired pressure, the retort is heated, vaporizing the oil under pressure. The oil-vapor is condensed in passing through the coil *D*, and the liquid distillate collects in the column $D^2$, and is drawn off, either continuously or intermittently.

chamber, *C*, which may be heated or partially exhausted. The pressure is regulated by a stop valve, *g*, a weighted check valve, *q*, being also provided. The unvolatilized liquid passes from the chamber *C* at *h*, and the vapor traverses a condenser, *H*.

As regards actual distillation under pressure, a number of early patents describe the manufacture of *illuminating oils* by the use of low pressures. Thus Young[1] stipulated pressures of 10 to 20 lb. per square inch. Krey[2] recommended 2 to 4 atmospheres and claimed to obtain a distillate of a specific gravity of 0.820 (yield not given). Boleg[3] recommended pressures of 3 to 4 atmospheres and stated that pressures of 4 to 6 atmospheres yield more "illuminating oil" but less "lubricating distillate" than the lower pressure. Dewar and Redwood[4] devised an

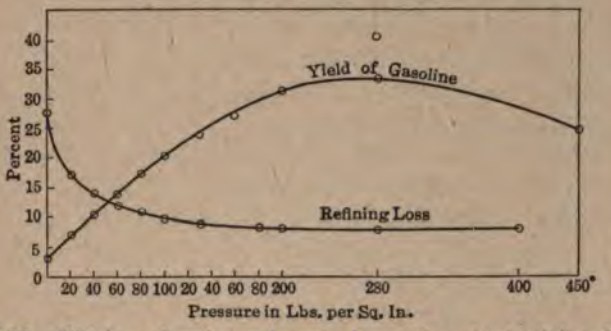



FIG. 197.—Yields and refining losses of gasoline obtained by distilling Oklahoma reduced oil under pressure. Gasoline = distillate below 150°C.; refining, by 1/10th volume concentrated sulphuric acid.

apparatus (see Fig. 196) for distilling petroleum under pressure, which is the general arrangement now employed on a larger scale by a later patentee. Dewar and Redwood, however, did not state *what pressure* gave the results desired, nor did they advocate their process as one suitable for manufacturing *gasoline*.

The foregoing brief review outlines the "state of the art" prior to the patent of Bacon and Clark.[5] The latter patent specifies the distillation of heavy petroleum oils between pressures of 100 and 300 lb. The advantages of the range specified, so

[1] English Patent 3345, Dec. 27, **1865.**

[2] German Patent 37728, **1886.**

[3] *Chem. Rev. Fett-Harz-Ind.*, **9** (1898), 24.

[4] United States Patents 419931, **1890**, and 426173, **1891**; English Patents 10277, **1889**, 13016, **1890**, and 5971, **1891.**

[5] United States Patent 1101482. Under Int. Conv., May 6, **1912.**

far as increased yields of gasoline are concerned, are brought out by the results represented graphically in Fig. 197. The yields given are not the maximum obtainable, but represent comparative results of a series of experiments obtained by distilling a given quantity of Oklahoma reduced oil at a *uniform rate* from the same apparatus at the different pressures indicated.

The effect of pressure in diminishing the per cent. of *olefines* in the gasoline obtained is a noteworthy feature of these results. The same effect is very strikingly shown in the recent results of Whitaker and Rittman[1] on the effect of pressure in the yield of illuminants in oil gas. At 900°C., Whitaker and Rittman obtained from a given quantity of oil 122 liters of illuminants at 0.75 lb. pressure, 50 liters at atmospheric pressure and 15.5 liters at 45 lb. (absolute) pressure. They were also able to show that at temperatures of 750° to 800°C. the addition of hydrogen to the gas mixture has the effect of partially hydrogenating the olefines, and that this reaction takes place more readily as the pressure on the system is increased. Ipatiew[2] has made the interesting observation that, in the distillation of petroleum under pressure, at the higher pressures the evolved gases become continually poorer in hydrogen in spite of the higher temperatures required to maintain the higher pressures. The pressures employed by Ipatiew were 120 to 340 atmospheres. Brooks, Bacon, Padgett and Humphrey found the following:

TABLE LI.—GASES FROM CRACKING DISTILLATIONS UNDER 100 LB. PRESSURE

| Sample number | (a) From Jennings crude | | | (b) Paraffin | | |
|---|---|---|---|---|---|---|
| | I | II | III | I | II | III |
| Temperature in still | 340°C. | 415°C. | 422°C. | 417°C. | 432°C. | 437°C. |
| $CO_2$ | 1.2 | 0.5 | 0.0 | 0.0 | 0.0 | 0.0 |
| CO | 1.2 | 0.5 | 1.3 | 0.0 | 37.0 | 33.5 |
| Illuminants | 15.4 | 15.3 | 13.0 | 25.4 | 0.0 | 0.0 |
| Hydrogen | 0.0 | 4.0 | 4.4 | 0.3 | 0.9 | 3.0 |
| Saturated hydrocarbons | 81.5 | 79.7 | 81.3 | 74.3 | 62.1 | 63.5 |

The analyses in Table LI. (*a*) were made of the gases evolved during a cracking distillation of a reduced oil prepared by removing from Jennings crude all constituents boiling below 265°C.

[1] *J. Ind. Eng. Chem.*, 6 (1914), 479.
[2] *Ber.*, 37 (1904), 2969.

Sample I was taken as soon as 100 lb. pressure had been reached. Sample II was collected after 40 per cent. of the charge had been distilled, and Sample III after 75 per cent. had distilled.

The liberation of hydrogen from petroleum hydrocarbons at various temperatures has been studied by Engler and his students.[1] They obtained no hydrogen below 470° at atmospheric pressure from *kerosene* fractions boiling below 280°C. The liberation of hydrogen from different hydrocarbons at a given temperature depends somewhat on their constitution. Thus, benzol yields appreciable quantities of hydrogen only at temperatures above 500°C.

The work of Brooks, Bacon, Padgett and Humphrey indicates that *if* hydrogenation of the liquid olefines takes place during distillation under pressure, it occurs simultaneously with their initial formation. A sample of cracked naphtha, having an iodine number of 55.0, was heated to 196°C. with hydrogen for 30 hr. under 3,000 lb. pressure per square inch. The iodine number and refining loss with sulphuric acid were practically unaffected, the iodine number of the final product being 52.9. Results closely parallel to these were obtained by Edmund O. Rhodes, in the laboratory of the Mellon Institute of Industrial Research, working with liquid fatty oils. The apparatus employed was a steel bomb connected with a solenoid stirrer constructed as described by Stucker and Enduli.[2]

Ubbelohde and Woronin[3] showed that in the presence of nickel, hydrogen was split off from a Baku crude oil at as low a temperature as 180°C. The results of Zelinsky[4] with platinum as a catalyst show that dehydrogenation at temperatures above 300°C. in the presence of platinum is to be expected. Ostromislenski and Buryanads[5] showed that in the presence of nickel a Russian crude oil gave only coke, 40 per cent., and gas at temperatures between 600–700°C., no tar or liquid being obtained at all. Furthermore, the gas contained 72 to 75 per cent. of hydrogen and the remainder consisted of saturated hydrocarbons. These facts are quite significant in view of the proposed cracking processes of Vernon Boys, Lamplough and others, who intro-

[1] Engler and Höfer, "Das Erdöl," **1**, 574.
[2] *Z. Elektrochem.*, **1913**, 570.
[3] *Petroleum*, **7** (1911), 9 and 334.
[4] *Ber.*, **45** (1912), 3678.
[5] *J. Russ. Phys.-Chem. Soc.*, **42** (1910), 195.

duce nickel into the cracking zone. A series of experiments were made by Brooks, Bacon, Padgett and Humphrey, at atmospheric pressure and at temperatures of 500–550°C., employing various catalytic substances. Kerosene and solar oil vapors passed through an iron tube containing burned clay, carbon, coarse and finely divided iron, coarse and finely divided copper, heated to the above-named temperature, yielded gasoline fractions, which showed an olefine content of approximately 25 to 30 per cent. When nickel was employed, the per cent. of olefines in the gasoline product was 48 per cent.

The *effect of nickel* in increasing the per cent. of olefines, when operating at atmospheric pressure, opens up the question of the effect of foreign substances on the polymerization of olefines and the effect thereon of different physical factors. The percentage of olefines indicated in the above figure does not represent the minimum obtainable even without the addition of a "catalyst."

The claim is made by Burton[1] that by placing the pressure-controlling valve beyond the condenser so as to condense the volatile gasoline vapors produced by distilling heavy petroleum oils under about 75 lb. pressure, olefines are not produced; whereas, if the pressure controlling valve is placed between the still and condenser, olefines are found in the condensate to such an extent as to render the latter process of doubtful commercial utility. Brooks and his co-workers felt that if olefines could somehow be squeezed together or polymerized by 75 lb. pressure in a condenser containing cold water, this fact would be an interesting contribution to science; but the experiment noted above, in which a cracked petroleum naphtha was heated for 30 hr. at 196°C. under 3,000 lb. pressure per square inch practically without change (iodine number reduced from 55.0 to 52.9), seemed to render such a reaction as Burton describes improbable. Brooks, Bacon, Padgett and Humphrey accordingly prepared several gallons of gasoline by distilling Oklahoma "reduced" oil at approximately 80 lb. pressure, in one case with the valve between the still and the condenser, and in another experiment with the still and condenser in free communication, so as to condense the vapors under pressure, all other conditions remaining the same.

[1] United States Patent 1049667. Under Int. Conv., July 3, 1912. The Burton process is described on p. 573.

| Gasoline | | |
|---|---|---|
| Per cent. refining loss by 5 per cent. conc. $H_2SO_4$... | 7.9 | 8.0 |
| Per cent. refining loss by 5 per cent. "oleum"..... | 12.2 | 12.0 |
| Kerosene, boiling point 302–392°F. | | |
| Per cent. refining loss by 5 per cent. "oleum"..... | 9.0 | 8.0 |

In I the valve was interposed between the still and condenser; and in II the still and condenser were in free communication, the control valve being beyond the condenser. These results seem to verify the opinion expressed above.

Dewar and Redwood[1] specified clearly that they condensed the distillate, obtained in their process, under pressure. In United States Patent 726173, they make the statement: "For this purpose we arrange a suitable boiler or retort and a condenser in free communication with one another, without interposing any valve between them; but we provide a regulated outlet for condensed liquid from the condenser." Aside from the above noted discovery as claimed by Burton, it is difficult to find any novelty in his patent. The operating pressures specified by Burton are 4 to 5 atmospheres. Boleg[2] described distillation under 4 atmospheres pressure, and in 1886 Krey[3] published the results obtained by distilling various crude oils, heavy oils and residues under pressures of 3 to 6 atmospheres. Engler[4] confirmed the claims of Krey, but, like Dewar and Redwood, Engler and Krey did not discover the remarkable effect of the position of the pressure-controlling valve, as referred to above.

It has been pointed out that the results presented in Fig. 197 do not represent the maximum yields of gasoline or the minimum percentage of olefines obtainable in the gasoline distillates made by distilling under pressure. Brooks, Bacon, Padgett and Humphrey did not point out the optimum working conditions of the process. However, in the patent of Bacon, Brooks and Clark[5] it has been shown that the ratio between the volume of the oil heated and the area of the heating surface of the apparatus employed is an important factor. This relation may perhaps be more clearly understood if it is stated that, other conditions remaining the same, cracking does not occur throughout the mass

[1] *Loc. cit.*

[2] *Chem. Rev. Fett-Harz-Ind.*, **9** (1898), 24.

[3] German Patent 37728, **1886.**

[4] *Ber.*, **30** (1897), 2919.

[5] United States Patent 1131309, Mar. 9, **1915.**

of heated oil, but that the *cracking effect produced in a given quantity, in a given time, will, within certain limits, be approximately proportional to the heated surface in contact with the oil.* It is obvious that distilling slowly will have approximately the same effect as increasing the relative area of heated surface. Another fact brought out in this process is that the deposition of coke on vertical heating surfaces is very much less, one-third

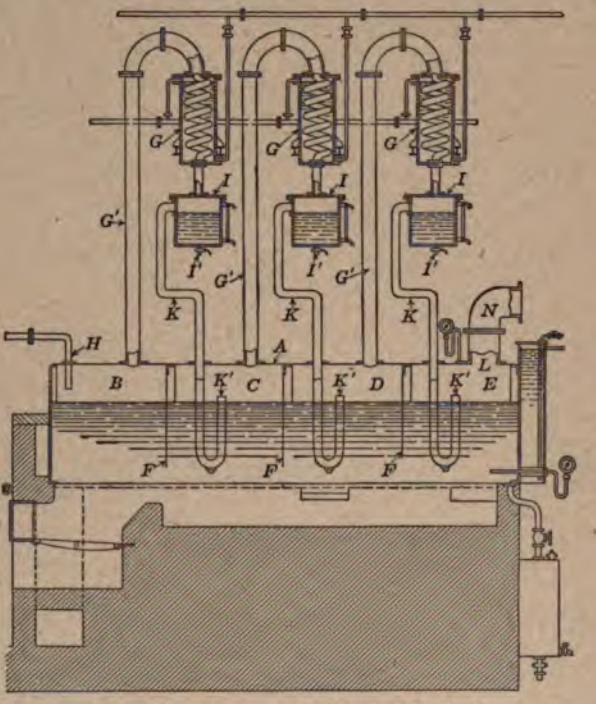



FIG. 198.—Apparatus patented by Laing.

The apparatus comprises a boiler divided into compartments (*B*, *C*, *D* and *E*) by partitions (*F*) not reaching to the bottom, each compartment being connected with a condenser (*G*) which delivers (*K*) to the next compartment. "To ensure steady working," a trap (*I*) is provided beneath each condenser, with means (*I'*) for drawing off water from the distillate, and the oil in the trap overflows through a down-pipe (*K*) which has a bend (*K'*).

to one-fifth of the amount deposited on the upper side of a horizontal surface, such as an ordinary still bottom, in a given length of time.

Under higher pressures, such as 200 lb. per square inch, oils having boiling points above 300° are almost completely prevented from distilling at the mean temperatures in the still, *i.e.*, 370° to 420°C.

Laing,[1] operating at much lower pressures, attempts to *return* the heavier boiling fractions of the distillate to the still by employing an apparatus such as is shown in Fig. 198.

Although hydrocarbons of the type contained in gasoline are more stable than the heavy hydrocarbons such as are contained in "reduced" oils, it is nevertheless important to remove the gasoline from the "sphere of reaction" as fast as formed. Thus, heating a certain volume of heavy oil under a given pressure for a given period of time, and subsequently distilling at atmospheric pressure, does not yield nearly as much gasoline as distillation under the same pressure, thus removing the gasoline and kerosene as fast as formed. This was well shown by an experiment cited by Brooks and his co-workers, in which Oklahoma "reduced" oil was heated under a pressure of 180 lb. per square inch for 2 hr. and then distilled at atmospheric pressure. There was obtained in this way only 5 per cent. of gasoline, while another experiment under the same conditions, except that the gasoline was distilled as fast as formed, yielded 30.4 per cent. gasoline. This indicates that the gasoline hydrocarbons themselves slowly decompose in the cracking still. In view of this fact, it is all the more remarkable that the kerosene fractions of the pressure distillate obtained from Oklahoma reduced oil by distilling under 100 lb. pressure are optically active.

**Optical Activity of Kerosene Hydrocarbons made by Cracking Heavier Hydrocarbons.**—The kerosene fractions of most crudes show very little, if any, optical activity. The maximum rotations are noticed in the fractions boiling from about 230° to 290°C. under 12 to 14 mm. pressure. A Galician oil studied by Engler yielded a fraction boiling from 200° to 250° under atmospheric pressure, which showed a rotation (200-mm. tube) of +0.2° on the saccharimeter scale. The fraction boiling from 200° to 250°C., made by cracking Oklahoma reduced oil under 100 lb. pressure, showed a rotation in a 400-mm. tube of +0.36° on the saccharimeter scale. Other samples showed rotations of approximately the same degree. In view of the high temperatures prevailing in the pressure still, 360°–420°C., the fact that complete racemization has not occurred indicates that these optically active hydrocarbons undergo racemization with greater difficulty than optically active compounds of any other

[1] German Patent 260858, October, **1911**; *Chem.-Ztg.*, **1913**, 375. See also English Patents 4120 of **1890** and 23446 of **1911**.

class. The corresponding fraction, boiling from 200°–250°C., distilled off the original Oklahoma crude, had a rotation of +0.2° in a 200-mm. tube on the saccharimeter scale. Since the optically active kerosene hydrocarbons made by cracking under pressure are to be regarded as products of the "splitting" of heavier hydrocarbons of higher molecular weight, the fact of their being optically active is even more remarkable. Engler and Bobrzynski[1] found that a fraction of Galician oil still showed an optical rotation of 0.8° (saccharimeter scale) after heating at 350°–360° for 4 hr. Engler has also shown that optically active oils may be obtained by the destructive distillation of cholesterol.

Engler[1] and Marcusson[2] have suggested that the optical activity of petroleum oils is due to hydrocarbons of the naphthene type. This is confirmed by observations that a viscous lubricating oil prepared from Jennings crude, after refining several times with "oleum" and finally clarifying with fullers' earth, showed an optical rotation in a 400-mm. tube of 4.04° arc or 11.6° on the saccharimeter scale. Since this oil was odorless and tasteless, optically active naphthenic acids could not have been a factor.

**Products of Cracking Distillation under Pressure: Formation of Benzene, Toluene, and Xylene.**—The formation of aromatic hydrocarbons from petroleum oils has heretofore been assumed to be due to complex and little understood changes, such as dehydrogenation of naphthenes, and the formation of acetylene and the condensation of the latter to benzene and its homologues.

Brooks, Bacon, Padgett and Humphrey found the above-named aromatic hydrocarbons in gasoline made by cracking heavy Oklahoma reduced oil under a pressure of 100 lb. per square inch. Treatment of the gasoline by liquid sulphur dioxide gave 10.0 per cent. of olefines and aromatic hydrocarbons, which mixture was fractionated several times and the fractions whose boiling points corresponded to the boiling points of benzol, toluol and xylol were nitrated in the usual manner, water added, and the crude crystalline mass thus obtained recrystallized several times from alcohol. They obtained in this way 1,3-dinitrobenzol, melting point 90.0°C., also 2,4-dinitrotoluol of

[1] ENGLER, "Das Erdöl," **1**, 218.
[2] *Chem.-Ztg.*, **35** (1911), 729.

melting point 70.0°C., and 2,4,6-trinitrometaxylol, melting at 182.0°C.

Since relatively little gas[1] is evolved when distilling under 100 lb. pressure, and since the gas evolved contains not more than 4.4. per cent. hydrogen, it appears highly improbable that the aromatic hydrocarbons in question are produced, either directly or indirectly, by a process of splitting off of hydrogen. Brooks and his co-workers believe *the evidence points to the splitting off of benzol, toluol and xylol from large so-called petroleum hydrocarbons, which contain the phenyl radical.*[2] In order to test this hypothesis further, they made "gasoline" by heating Oklahoma reduced oil with anhydrous aluminum chloride. In this reaction very little gas is evolved from dry petroleum oils. Should the phenyl radicals exist in the complex petroleum hydrocarbons, one might expect a splitting of the molecule, just as is known to occur when toluol is treated with aluminum chloride,

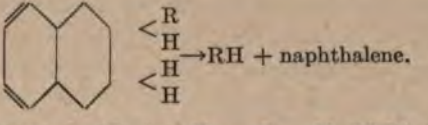
$$C_6H_6 \leftarrow C_6H_5.CH_3 \rightarrow C_6H_4{<}^{CH_3}_{CH_3},$$

yielding both benzol and xylol.

Twenty liters of Oklahoma reduced oil were slowly heated with 6 per cent. anhydrous aluminum chloride until 4 liters of light distillate were collected. This was treated with liquid sulphur dioxide, 0.40 liter of sulphur dioxide soluble extract being ob-

[1] This will vary with different oils and the evolution of gas becomes much more rapid toward the end of a distillation when much coke is produced.

[2] Jones and Wheeler (*J. Chem. Soc.*, **1914**, 2562) have shown that hydrocarbons of the aromatic series are produced in the distillation of coal at 350°C., or below the temperatures at which hydrogen is split off. They suggest that aromatic compounds may be formed by reactions such as the following:




Brooks and Humphrey (*J. Am. Chem. Soc.*, **38** (1916), 393) have found benzene, toluene, and *m*-xylene among the products from the cracking of heavy petroleum oils and conclude that the heavy high-boiling fractions of petroleum contain homologues of benzene, or hydrocarbons containing the benzene nucleus.

On the effect of temperature on the formation of benzene from petroleum at atmospheric pressure, see Egloff and Twomey, *J. Phys. Chem.*, **20** (1916), 121.

tained. This was fractionated three times and the proper fractions nitrated in the usual manner. Metadinitrobenzol, 2,4-dinitrotoluol and 2,4,6-trinitro-metaxylol were easily obtained in a state of purity after recrystallizing several times from dilute alcohol.

There are, of course, other conditions in which aromatic hydrocarbons are formed by complex reactions carried out at much higher temperatures attended by the evolution of large quantities of hydrogen and unsaturated hydrocarbons, as in the manufacture of oil gas. These conditions, however, are very different from those prevailing in the above experiments.

Since practically all of the crude petroleums of the world which have been examined, yield gasolines and kerosenes containing small percentages of aromatic hydrocarbons, and in view of the commonly accepted theory of the origin of petroleum from animal and vegetable remains, it is to be expected that hydrocarbons of high molecular weight containing the phenyl radical are to be found in heavy petroleum oils. Such hydrocarbons, containing aromatic radicals, cannot be formed by any simple anaerobic degradation of fats, but probably, like the nitrogen bases, have been derived originally from protein material. We may therefore expect to find relatively large percentages of *aromatic compounds in petroleums that are also rich in nitrogen.* This appears to hold true in the case of the California petroleums, which are high in nitrogen, and, according to work carried out in the Mellon Institute of Industrial Research, this is also true of certain heavy Mexican oils. On the other hand, it may be pointed out that the oils of Borneo, Sumatra and Java are relatively rich in light aromatic compounds, but low in nitrogen. The latter oils, however, are very light and fluid and very different from the heavy pitch-like oils of California and Mexico, and it has been frequently suggested that these light fluid oils have been purified by filtration through porous strata. Such light, supposedly filtered, crudes are low in sulphur as well as nitrogen. Thus the converse of the above statement does not hold, probably for the reasons just indicated.

Another noteworthy feature of gasoline made by cracking at a pressure of 100 lb. is that *normal paraffin hydrocarbons* appear to be the predominating constituents. The amount of isopentane exceeds that of normal pentane, but the hexane and heptane fractions appear to consist chiefly of the normal paraffins. These

facts are brought out by Fig. 199, representing the results of seven fractional distillations.

| Per cent. | Found | Calculated For $C_6H_{14}$ | Calculated For $C_6H_{12}$ |
|---|---|---|---|
| Carbon | 85.06 | 83.72 | 85.72 |
| Hydrogen | 14.93 | 16.28 | 14.28 |

Only a very small amount of naphthenes appears to be present. Some evidence of the presence of methyl cyclopentane was shown by the above combustion analysis of the fraction boiling from 68° to 70°C.

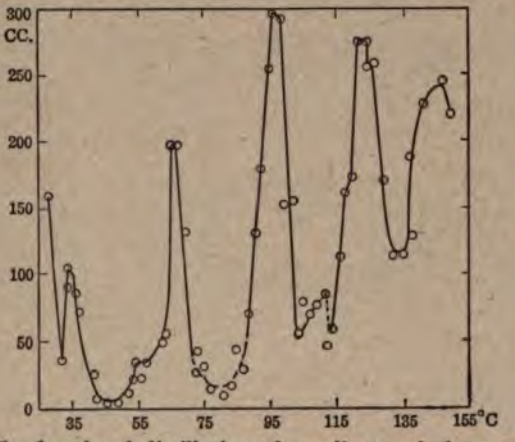



FIG. 199.—The fractional distillation of gasoline made by cracking under 100 lb. pressure.

**The Burton Process.**—The process of W. M. Burton, which is being used on a large scale for the manufacture of "motor spirits,"[1] the name under which the gasoline so made is being put on the market, relates to the treatment of high boiling-point residuals, particularly the residuum from the distillation of paraffin-base petroleum, after the lower boiling distillates are removed, in order to obtain a low boiling-point product of the paraffin series, avoiding the ethylene series. This is accomplished

[1] This product averages about 60°Bé. gravity, has a pronounced odor and pale straw color, and is, it is claimed, composed principally of hydrocarbons of the paraffin series. It is said to be equal to gasoline (see p. 481) for motor use (T. T. GRAY, *Min. Ind.*, **21**, 657). It is reported that the production of "motor spirits" by the use of the Burton process now (April, 1916) amounts to over 3,000,000 bbl. per year. On the Burton process, see also *Nat. Petrol. News*, **5**, No. 5, 1; and *Sci. Am.*, **112** (1915), 5. The patent of BURTON is discussed on p. 566.

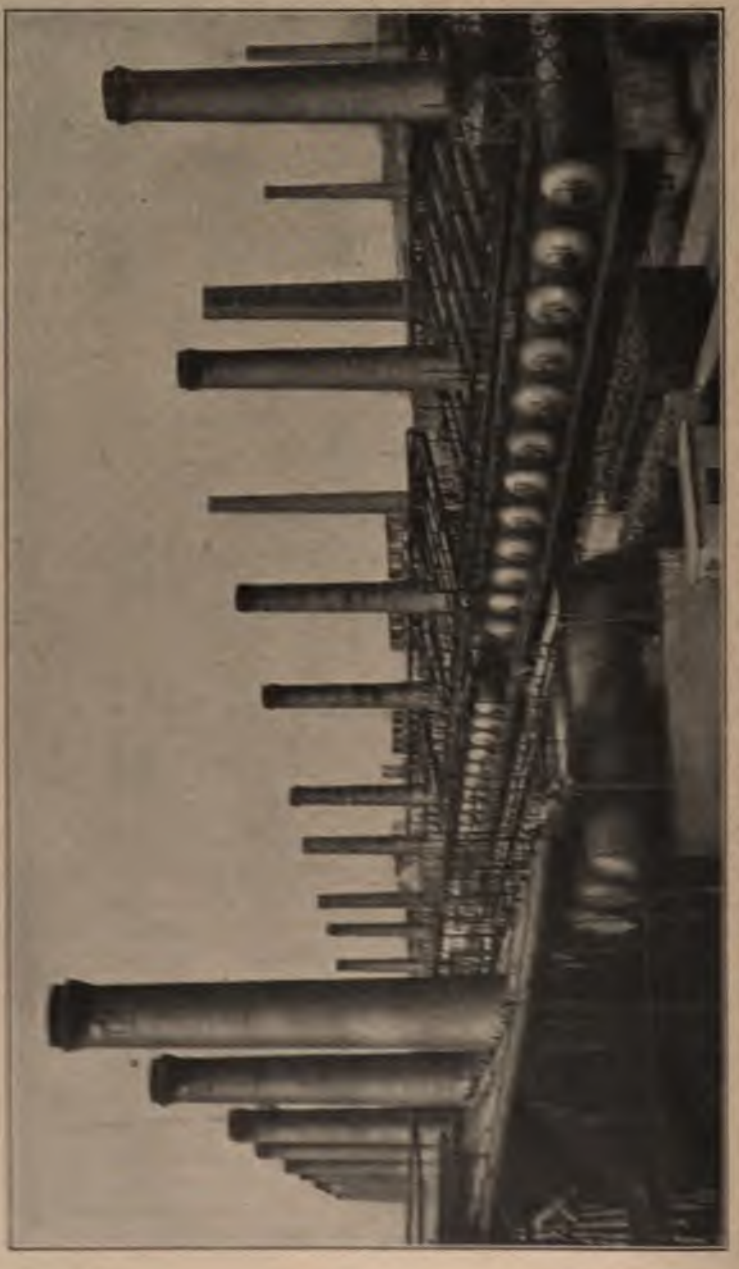


FIG. 200.—Plant for the production of gasoline by the Burton process.

by raising the boiling point of the liquid residue by means of back pressure on the liquid and vapors, which are maintained under pressure throughout their course from the still through the condenser, and while undergoing condensation.

The apparatus used by Burton consists of a boiler equipped with a safety valve, fireplace, pressure gage and thermometer. A pipe leads from the top of this boiler to a condenser, consisting of a pipe-coil immersed in the usual cooling tank. From near the lower end of this coil, a small pipe leads up to a relief valve, and lower down, connecting to a receiver for the products of condensation, is a shut-off valve. This valve is normally closed. The relief valve allows any excess gas pressure to escape. The pressure with the shut-off valve closed, is from 4 to 5 atmospheres, raising the boiling point from 500° to 750° or 800°F. This pressure combined with the temperature, greatly increases the conversion of the high-boiling members of the paraffin series into the low-boiling members of the same series.[1]

Although the arrangement of Burton's apparatus is essentially the same as that of Dewar and Redwood,[2] and the pressure used by him, 75 lb. per square inch, had been previously employed by others, Burton has been the first to demonstrate that such distillation can be safely carried out on a large scale. His process is in use by the Standard Oil Company and plants have been erected at a number of points.[3]

**The Rittman Process.**—In the process of Walter F. Rittman[4] vaporized petroleum is passed into a hot tube at 450°C., the pressure being 90 to 500 lb. per square inch; the vapors are condensed under pressure and the condensate is distilled for its gasoline content. The residue may be revaporized and passed through the tube. The following claims are made for this process: By using the oil in the form of vapor any combination of temperature and pressure may be applied; any oil from kerosene up may be used; in the case of fire, only a small amount

[1] United States Patent 1049667, Jan. 7, **1913.**

[2] See p. 562.

[3] One of these, at Whiting, Ind., was constructed at a cost of $700,000.

[4] *Nat. Petrol. News*, **7** (1915), 2. For a detailed account of the Rittman process, see *Bull.* **114**, *Bureau of Mines*, **1916**; and *J. Ind. Eng. Chem.*, **8** (1916), 351. On the differences between the process of Rittman and that of Snelling, wherein the temperatures and pressures are limited, see WILLIAMS, *Bull. Am. Inst. Min. Eng.*, 1916, No. **109**, 173. On the history of the Rittman process, see *Oil, Paint and Drug Rep.*, **89** (1916), No. 8, 7.

of oil is exposed; and by reason of the reaction being gaseous instead of liquid, the carbon deposited is so small as to be negligible.

Rittman has recently described experiments, the results of which have indicated the following conclusions:[1]

1. Equilibrium products of the cracking reaction seem to be independent of the influence of chemical and physical properties of the original oils or at most are affected only in minor degree by such tendencies. The matter of carbon formation is the one clear-cut exception. Carbon is a residual product and its formation is proportional to the amount originally contained in the oil. Viscosities and specific gravities seem to show in slight degree the influence of properties of original oils, but the differences are so slight that they may well be explained on the basis of failure to reach complete equilibrium.

2. The formation of aromatic hydrocarbons occurs best at moderate to high temperatures and under high pressure.[2]

3. The formation of gasoline (low-boiling, low-gravity hydrocarbons) is favored by moderate to low temperatures and by high pressure.

**The McAfee Process.**[3]—As indicated above, in most of the cracking processes, pressure is employed: 60 to 100 lb. is usual and some have proposed much higher pressures. It is here that the greatest difficulty becomes manifest—the great danger to the operators and to the plant. There is always a deposition of hard (and flinty hard) coke on the inner walls of the heating element. Some who have had considerable experience in cracking oils have said that the coke is forced into combination with the iron, making it brittle and thus utterly unable to withstand the high temperature and pressure employed. At any rate, the deposition of the carbon occurs where the element is hottest, causing a local overheating at that point. Under such conditions the tensile strength of the steel becomes an unknown

[1] *J. Ind. Eng. Chem.*, **7** (1915), 945.

[2] On the equilibrium relations among aromatic hydrocarbons produced by cracking petroleum, see RITTMAN and TWOMEY, *J. Ind. Eng. Chem.*, **8** (1916), 20.

[3] *Nat. Petrol. News*, **7** (1915), 20; and especially *J. Ind. Eng. Chem.*, **7** (1915), 737.

The McAfee process is owned by the Gulf Refining Company. It is not a "cracking process," but it is included in this section because of its application.

quantity; as likely as not it yields to the stress without any warning. Oil vapors of a temperature around 650°F. ignite spontaneously when they evolve from a still into the surrounding atmosphere. In cracking processes the temperature is 750° to 850°F. and even higher; hence, the manifest danger of cracking petroleum oils under such pressures.

When McAfee took up the gasoline problem, he dismissed from consideration the idea of cracking oils. Since the Friedel and Crafts English Patent, No. 4769 of 1877, there had been some degree of mild interest shown as regards the effect which aluminum chloride might have on petroleum oils, but no positive results had followed from this interest. It appeared to McAfee that this reaction might have far more significance than was apparent.

McAfee found that with proper control of the vapors leaving the distilling system and entering the final condenser, and with sufficient time given the aluminum chloride, high-boiling oils can be completely broken down into lower boiling oils, and no matter how unsaturated the high-boiling hydrocarbons may be, the low-boiling oils produced therefrom are sweet smelling, water-white and saturated. The reaction gives little gas and only about the right amount of carbon to allow production of saturated products. The carbon is deposited, not in the form of a hard baked-on carbon, but as a granular coky mass, easily removed from the still.

The problem before McAfee, knowing the reactive power of aluminum chloride, was to apply it to crude petroleum so that good heavy oils could be obtained while, at the same time, converting the less valuable portions of the crude into gasoline. The solution of this problem is found in United States Patent 1127465 of Feb. 9, 1915.[1]

In the practical operation of this process, crude petroleum of any kind is first distilled until the naturally occurring gasoline and kerosene, if there be any present, are distilled off. In many crude oils, and especially some of those from Texas, California and Mexico, there is substantially no gasoline present and very little kerosene. But in any event, the crude is first heated to free it of any moisture which it may contain, since the oil must be perfectly dry before adding the aluminum chloride.

[1] See also Canadian Patents 163091, 163092, and 163093, June 8, 1915; and 163671, July 6, 1915.

The next step is to add anhydrous aluminum chloride to the remaining residual oil, and then bring the mixture to boiling in the still. Boiling is usually around 500°F. and generally remains between 500° and 550°F. during the entire distillation, extending over a period of 24 to 48 hr. There is no need of employing extra pressure or vacuum or special apparatus; any still with a stirrer in it suffices.

Granted sufficient time for the aluminum chloride to produce effects, the success or failure of this process depends upon the proper control of the temperature at which the oil vapors are allowed to leave the distilling system to enter the final condenser. Between the still and the final condenser are placed two air-cooled condensers connected in series, which separate the low-boiling oils from the high-boiling oils, returning the latter, together with any volatilized aluminum chloride, to the still. For a 1,000-bbl. still the air condensers which are now used are drums of oval cross-section, 3 ft. by 6 ft. by 6 ft. high. In addition to the air condensers, a 3-ft. dome is attached to the top of the still which serves to return most of the volatilized aluminum chloride and its compounds. The operation is so controlled that the vapor is kept at the desired temperature as indicated by a thermometer placed in the vapor line at the point of exit of vapor into the final or water-cooled condenser. The temperature at this point should not exceed 350°F.; otherwise, not only will heavy oils distil over, but the aluminum chloride (or its compounds with hydrocarbons) will enter the condenser and clog it up. Under the first-named condition, the distillate obtained will be a mixture of gasoline, solvent oil and kerosene, which are afterward separated by fractional distillation. These products are all water-white, sweet smelling, saturated, and need no refining with sulphuric acid. In practice, no treatment is given them, except a washing with alkali, followed with water, to remove hydrogen sulphide. With proper back-trapping of high-boiling oils into the still from the air-cooled condensers and a temperature of 300°F. in the vapor line, the distillate obtained will be gasoline alone, which is ready for the market when washed with an alkaline solution.

While the operation, using crude oil as the starting material, can be carried on to produce larger or smaller quantities of gasoline, in practice it is carried on so as to convert the gas-oil fraction into low-boiling hydrocarbons and leave most of the

high-boiling hydrocarbons, that is, the paraffins and lubricating oils. Accordingly, the operation is interrupted after a portion of the crude has been converted into low-boiling products, and the high-boiling oil remaining in the still is pumped off while hot; on cooling it is worked up into the usual paraffin and lubricating products. The aluminum chloride remains in the still enmeshed in a mass of coke, and the methods of its recovery are found in United States Patents 1099096, June 2, 1914, and 1144304, June 22, 1915.

**The Deodorization of "Cracked" Gasoline.**—It has been found at the Mellon Institute of Industrial Research that highly "cracked" gasoline loses nearly all of its offensive "cracked" odor when treated with alkaline plumbite, cupric oxide, or sodium.[1] It appears from certain investigations carried out at the Mellon Institute that the offensive odor of "cracked" oils is due to the presence of small quantities of sulphur compounds, naphthenic acids, and basic nitrogen compounds, the latter probably of the pyridene type.

## THE CHEMICAL TREATMENT OF PETROLEUM DISTILLATES

The methods adopted for the separation from petroleum of various products of commercial importance, and their purification by chemical treatment, have been described in the preceding chapter in more or less general terms. The chemical treatment of the distillates of petroleum will now be discussed in some detail.[2]

**Treatment with Sulphuric Acid.**—Most of the products obtained by the distillation of petroleum are not in a marketable condition, but require *chemical treatment* to remove the resinous matters and the hydrocarbons of the unsaturated and aromatic groups, which impart a dark color as well as an unpleasant odor to the liquid, and, in the case of illuminating oils, increase the

[1] On freeing cracked mineral oils from malodorous or resinifying substances by treatment with a salt of a heavy metal having a condensing action, see English Patent 3572, Feb. 11, **1914**.

[2] No consideration is given here to the preparation of anthracene, etc., from petroleum residuum (see REDWOOD, *J. Soc. Chem. Ind.*, **4** (1885), 70), to the production of aromatic bodies from petroleum (see *Dingler's polyt. J.*, **229** (1878), 353; LIEBERMANN and BURG, *Ber.*, **11** (1878), 723; and SALZMANN and WICHELHAUS, *idem*, **11**, 802 and 1431), or to the preparation of benzene and its homologues (see OGLOBLIN, *Z. Farben- u. Textil-Ind.*, **3** (1904), 293). See also pp. 570, 576 and 806.

smoke-producing qualities and reduce the power of rising in the wick by capillary attraction.[1]

During the primary development of the petroleum industry, kerosene was marketed as a yellow or dark-red liquid. The removal of color was attempted by treatment with soda lye and lime; but it was found that after oil so purified had been burned for a short time, the wicks became encrusted, and consequently the oil failed to rise properly. Subsequent treatment with hydrochloric acid was tried, but this effected very little improvement. Sulphuric acid had been employed in the "coal oil" industry, especially by Joshua Merrill[2] in the refining of keroselene[3] in 1857, and the refiners of petroleum early profited from the experience of the coal oil manufacturers and adopted treatment with sulphuric acid, followed by that with soda lye. This process is in general employment at the present time.[4] The rationale of this treatment is not fully understood, but the action appears to consist in the removal or decomposition of the aromatic hydrocarbons, fatty and other acids, phenols, tarry products, some sulphur compounds, etc., which lower the quality of the oil, the sulphuric acid removing some, while the sodium hydroxide removes the remainder and neutralizes the acid which has been left in the oil.

K. I. Tumski has discussed the action of sulphuric acid.[5] He states that the petroleum loses and the acid gains in volume, often to 5 to 8 per cent. of the petroleum; that the tar products dissolve and impart a deep brown color to the acid, while the aromatic hydrocarbons, and many of the other unsaturated hydrocarbons,[6] are converted into sulpho-acids; and that the acid is instrumental in removing the oxygen-containing bodies—in fact, according to Tumski, by continuing the treatment, the

[1] See section on *refining petroleum by liquid sulphur dioxide* (p. 593) for further information on the chemical treatment of illuminating oils.

[2] See HAYES, *Am. Chemist*, **2** (1872), 402.

[3] See p. 878.

[4] See pp. 460, 465, 467 and 473. For general descriptions of the chemical process of refining, see SCHESTOPAL, *Chem.-Ztg.*, **15** (1891), 352; VEITH'S "Das Erdöl," **1892**; and GURWITSCH'S "Wissenschaftliche Grundlagen der Erdölbearbeitung," **1913**, part ii. EICHLER, of Baku, was the first to introduce, in Russia, treatment with sulphuric acid, followed by that with caustic soda.

[5] "Technology of Naphtha" (Russian, Moscow, **1891**), 204.

[6] Cf. THIELE, *Chem.-Ztg.*, **37**, 841.

petroleum can be completely deprived of oxygen. However, the action of the sulphuric acid is more complicated than appears at first sight, for sulphur dioxide is evolved in some quantity, and this is not accounted for by the reactions referred to. Reduction of the acid evidently occurs, but the reducing agent is unknown.[1] It has been suggested that the acid has also an oxidizing action,[2] and this view is rendered partial support by the increased action when the treatment is assisted by heat, and by the greater refining power of "oleum" (Nordhausen or fuming sulphuric acid) as compared with the ordinary acid. The acid generally used is sulphuric acid having a gravity of 168°Tw. or 66°Bé. (1.840 specific gravity), but, occasionally, particularly where the oils are difficult to refine, oleum is used on account of its more energetic action,[3] and the use of sulphuric anhydride has been proposed in some cases.[4]

The quantity of acid required, and the length of time during which the treatment is continued, depend on the grade of product desired and on the purity of the distillate operated upon; the last-mentioned factor in turn depends on the composition of the crude oil and on the manner in which the distillation has been conducted. "Cracked" oils require a larger amount of acid than uncracked oils, and Canadian oil requires more acid and more prolonged treatment than Pennsylvania oil. Usually the amount of acid required increases in proportion to the density of the distillate. Oils long exposed to the air, and overheated distillates, require more acid and longer treatment than usual. Some investigators state that sulphuric acid causes polymerization of the hydrocarbons; but it is found that an increase in the amount of acid, or in the time over which the action extends, results in the production of a lighter oil, the removal of heavy tar products more than counterbalancing the increased density due to polymerization, if such change takes place.

Edeleanu and Gané[5] observe that in petroleum refining one

[1] Even when the acid treatment is repeated as often as ten times, color is still imparted to the acid, and further evolution of sulphur dioxide occurs.

[2] On the oxidizing action of sulphuric acid on mineral oils, see Holde, *Chem.-Ztg.*, **19** (1895), 144.

[3] Sand or other inert material has been used with sulphuric acid, to give it greater surface and in that way increase its activity; but some experts maintain that the chemical activity should be diminished.

[4] On the employment of sulphuric anhydride in refining, see p. 605.

[5] *Oesterr. Chem. Tech. Ztg.*, **26**, 156.

obtains the formation of sulpho-acids,[1] polymerization, oxidation,[2] or decomposition of the hydrocarbons, according to the temperature and the concentration of the sulphuric acid used.

Schulz[3] states that the color, opacity and fluorescence of petroleum are due to the presence of an oleosol of high molecular weight, probably containing oxygen, sulphur and nitrogen. Although chemical processes aid, in his opinion the real mechanism of refining is the precipitation of the resinous pitchy and asphaltic materials which exist in colloidal solution in the oil. Schulz found that with up to 3 per cent. of 93.72 per cent. sulphuric acid, the more acid used in refining, the better were the results obtained; that weaker acids gave poorer results; and that fractional refining gave no better results than adding the acid all at once.

Condrea[4] considers that refining by sulphuric acid is due to the polymerization and oxidation of colored decomposition products formed in distillation, and the solution of the resulting compounds in the acid. He maintains that it is not to be attributed to the precipitation of colloidal asphaltic material.[5]

Von Pilat and Starkel[6] noted wide differences between the results obtained by titrating sludge acids with alkali and those found by determining the acid content gravimetrically as barium sulphate. Since no aromatic sulpho-acids could be isolated from the filtrate of barium sulphate, these chemists concluded that such acids are not formed under the conditions existing in refineries.[7] They expressed the opinion that these acid compounds are either addition products of sulphuric acid residues with unsaturated hydrocarbons or substitution products of sulphuric acid through organic radicals, and concluded that sulphuric acid acts chiefly as a polymerizer.

[1] Sulpho-acids are decomposed and hydrocarbons are formed when they are distilled with superheated steam. For a process of producing sulpho-acids from petroleum, see H. A. Frasch, English Patents 10321 and 10322, May 25, 1893.

[2] On the oxidation of sulphuric acid, see Holde, *Chem.-Ztg.*, **19** (1895), 144.

[3] *Petroleum*, **5**, 205 and 446.

[4] *Rev. Gén.* (*Bucharest*), **5** (1900), 83.

[5] Cf. Schulz, *supra*.

[6] *Petroleum*, **6**, 2177.

[7] They did not deny, however, that aromatic sulpho-acids might be formed when oils were treated with oleum, at, say, 80°C.

Hausmann[1] has investigated black, regenerated sulphuric acid, obtained in the refining of petroleum in the provinces of Campani. He found that in order to obtain a good soda test in the oil treated with regenerated acid, an amount of sodium hydroxide much in excess of the theoretical must be used. This he explained by the presence of an acid weaker than the naphthenic acids, carried into the oil by the black acid. Oil treated with sulphuric acid in which the sulpho-acids from a sludge acid were dissolved, gave a poor soda test.

**The Effect of Impurities in Sulphuric Acid.**—The examination of sulphuric acid for petroleum refineries has been discussed by Ferdinand Schulz,[2] who points out that nitrogen trioxide ($N_2O_3$), nitrogen pentoxide ($N_2O_5$), and selenium dioxide ($SeO_2$) will cause discoloration of the refined oils. We have it on the authority of Schulz[3] that the presence of selenious acid in the sulphuric acid used in the refining of petroleum causes the oil to assume a yellow color which is not removable by subsequent treatment with sulphuric acid. He also states that sulphuric acid containing over 0.005 per cent. of selenium dioxide turns black when agitated with refined mineral oil. According to Kharitchkov,[4] the color of illuminating oil refined by treatment with sulphuric acid is very injuriously affected by the presence of nitrous acid, of which as little as 0.02 per cent. produces a marked effect. The presence of nitric acid is also objectionable, but 0.05 to 0.07 per cent. may be present without producing serious injury. The small amount of organic matter which may be present in commercial sulphuric acid is said to be without appreciable effect.

Acid storage tanks in refineries should be subject to periodical cleaning.

**Physical Factors Involved in Treatment with Sulphuric Acid.**—Zaloziecki[5] maintains that the best refining results are invariably obtained by conducting the acid treatment at as low a temperature as possible, for oxidation increases rapidly with a rise of temperature. He therefore advises[6] that the oil be pumped into agitators over night to cool, and that the acid treatment be

[1] *Petroleum*, **6**, 2301. See also *idem*, **7**, 13.

[2] *Chem. Rev. Fett- Harz-Ind.*, **20**, 82.

[3] *Chem.-Ztg.*, **35**, 1129.

[4] *Westnik shirow. weschtsch.*, **6** (1905), 119.

[5] *Chem.-Ztg.*, **19** (1895), 78. On low-temperature acid treatment, see also Guiselin, *Petroleum*, **6** (1910), 133.

[6] *Ibid.*, 875 and 956.

performed in the early morning; if artificial cooling is necessary, a refrigerating coil should be used. Zaloziecki also urges that light be excluded from the agitators.

Zahler[1] objects to the assertion of Zaloziecki, that the acid treatment should be conducted at the lowest possible temperatures, pointing out that each distillate has its own suitable temperature for treatment with sulphuric acid. Since, however, the refining value of sulphuric acid is largely due to its solvent action, and chemical action should be avoided as much as possible, the authors consider that Zaloziecki is correct in his contention.

According to Zahler, the form of the agitators[2] used, the duration and mode of mixing, the purity and strength of the sulphuric acid, all influence the color of the treated distillates of petroleum, and an increased temperature may, by facilitating the deposition of the "sludge acid," shorten the process and thus prevent the deepening of color so often observed in the upper layers of an oil after prolonged standing in the agitator.

Then, too, the time of the year has also to be considered. In summer, the chemical treatment should be effected at lower temperatures than in winter, because the heat evolved by the interaction of the oil and sulphuric acid may, especially when an air blast is used, rise beyond suitable limits; whereas in winter, a certain degree of warmth is necessary to assist the precipitation of the sludge, especially in the case of the heavy oils.

Experiments by Salomon[3] on the effect of air, of different degrees of purity, in the usual air-blast agitators,[4] have shown that by the use of purified air the amount of sulphuric acid required to refine petroleum distillates may be reduced. According to Salomon's plan, the air to be purified is passed through waste sulphuric acid from the agitators.

**The Recovery of Sulphuric Acid.**—The waste acid and alkali were formerly regarded as of but little value, although the latter was, in some cases, employed in the manufacture of common soaps. The sludge acid was used in the manufacture of artificial

[1] *Ibid.*, 215.

[2] See pp. 744 to 753. Of interest in this connection is the process of E. D. KENDALL (United States Patent 1154516, Sept. 21, **1915**), wherein sulphuric acid is mixed with the oil to be refined and the mixture is spread upon balls of glass, porcelain or stoneware which are tumbled in rotating drums.

[3] *Trudy Bak. otd.*, **1899**, 1; *Oesterr. Chem. Tech. Ztg.*, **17**, 3.

[4] See p. 751.

manures,[1] or, where its cost was great, it was sometimes diluted with three or four volumes of water, drawn off from the oil which was thus separated, and concentrated in iron or leaden pans to 60°Bé. for use for various purposes. In many cases the acid from the refining of naphtha was employed for the first treatment of kerosene, and the last portion of acid used for the kerosene was used for refining other oils. It is now customary to recover the acid,[2] and a number of processes have been devised for the purpose of carrying out this recovery.[3]

**European Practice in the Regeneration of Sludge Acid.**—Continental refiners divide sludge acid into two groups: (1) the sulphuric acid sediment from the refining of benzine and illuminating oil; and (2) that from the refining of lubricating oils. In the former, 10 per cent. of the acid is consumed, being either decomposed or combined; 100 parts by weight of unused acid of 66°Bé. yield 125 to 130 parts of 50° to 55°Bé. sludge acid, containing in solution asphaltic and sulphurous organic matter. By diluting this acid to 35°Bé., most of the oily matter (20 to 25 per cent. by volume) is separated. The bottom layer consists of dilute sulphuric acid, which requires concentration.

Since the concentrated acid is generally black and contains undesirable organic compounds, various methods have been proposed for its clarification.[4] Diffusion through a porous diaphragm against water gives a clear acid, but organic and sulphoacids are also carried along, and, on the evaporation of the acid,

[1] In 1872, sludge acid was generally sold in the United States to fertilizer manufacturers. KOCHETKOV (*Izv. Moskov. Selsk. Khoz. Inst.*, **19**, 69) has discussed the preparation of superphosphate with the waste sulphuric acid from petroleum refineries. With bone meal this waste gave superphosphates containing 21 per cent. of phosphoric acid, all water-soluble. With rock phosphate, 14 per cent. of phosphoric acid, somewhat less soluble, was obtained. On the use of regenerated sulphuric acid in the manufacture of superphosphate, see also MENNICKE, *Z. angew. Chem.*, **13** (1900), 1031.

[2] As late as 1911, however, waste acids from the German paraffin oil industry were diluted to 1 per cent. of free sulphuric acid and then delivered into the channel of an arm of the Elbe River at Hamburg (GÖHLICH, *Apoth.-Ztg.*, **26**, 420).

[3] On regenerated sulphuric acid, see HEINRICI, *Z. angew. Chem.*, **11** (1898), 525; MEYER, *J. techn. Chem.*, **17** (1833), 87; OSTREJKO, *Naphta*, **10** (1902), 258; PILAT and STARKEL, *Petroleum*, **6** (1911), 2177; SCHEITHAUER, *Jahresber. tech. Ver. sachs-thur. Min. Ind.*, **1888**; and WEDGE, *J. Soc. Chem. Ind.*, **18** (1899), 345. Attention is particularly directed to the early paper of MEYER and to WEDGE'S report on concentration experiments.

[4] See, in this connection, WISPEK, *Petroleum*, **6**, 1045.

are decomposed, turning the acid black. Regeneration by means of silicon compounds yields a yellowish acid, which becomes black when evaporated. Filtration through bone-black and heating with nitric acid is also unsuccessful. Evaporation in lead pans to 60°Bé. and then in glass or porcelain dishes to 66°Bé., has been generally used, but much sulphur dioxide is evolved and the acids foam considerably.

Wispek[1] states that the best method is that of Balcher and Stenzel, in use in Rumania. In this procedure for the regeneration of the waste sulphuric acid of refineries,[2] the acid is mixed with 50 per cent. by volume of water,[3] and then concentrated to 60°Bé. in lead pans. From the last pan it runs into a distillation retort, the lower part of which is of cast iron, the upper of volvic lava, and is then heated to 300°C. to destroy the organic matter. A current of air is pumped through the acid, when decoloration occurs; the 30°Bé. acid vaporizes, and clear concentrated sulphuric acid passes out of the retort. The volumes of the incoming and outgoing acids must be equal, so that the concentration of the acid in the retort should not change.

In the Baku region[4] the sulphuric acid that has been used in the refining of petroleum, is frequently diluted to 35° to 37°Bé. and warmed, when most of the organic matter is separated as a resinous mass. The acid is then concentrated to the carbonization of organic matter, again diluted to 36°Bé. to separate more organic matter, and finally concentrated to 66°Bé. However, the acid used in refining heavy asphaltic oils cannot be thus regenerated. The thick tar acids are washed, after evaporation by steam, and then concentrated to such a consistency that they can be mixed with natural asphalt to soften it.

The milky waste liquors from the refining of illuminating and lubricating oils are said to be utilized in the following manner at Baku:[5] The strongest acid runnings are worked up for acid, while most of the other waters, acid and alkaline, are so run together that they neutralize each other and the oils are caught on the surface. The oils from the refining of kerosene are used

[1] *Loc. cit.*

[2] *Petrol. Rev.*, **25**, 195.

[3] To separate 20 to 25 per cent. of the oil.

[4] WISCHIN, *Oesterr. Chem. Tech. Ztg.*, **26**, 147. On the utilization of the waste products from Russian refineries, see also MARKOVNIKOV, *Petrol. Ind. Techn. Rev.*, **3**, *Suppl.*, 41 (1900).

[5] PYHÄLÄ, *Petroleum*, **3**, 571.

for low-grade burning oils and as adulterants in soap-making. The spent liquors from the making of lubricating oils give rise to oils that are much used for soap filling or mixing with fats to make low-grade soaps. Siccatives, varnishes, greases, and electrical insulating materials have also been made from these oils.[1]

Russian experience has shown that dilution, air, and light are the three principal factors involved in the decomposition of sludge acid. In purifying the separated acid, Russian petroleum chemists have found that an addition of 0.5 to 1.0 per cent. of bleaching powder or potassium chlorate is efficacious, but we are not aware that such a procedure has been used in practice. By allowing the sulphuric acid to react at high temperatures on air-dried fire clay, Zaloziecki[2] obtained light-colored solutions free from organic matter and utilizable for making aluminum sulphate or alum: this method has decided limitations and is not in use, so far as the authors can learn.

Several patented processes for the regeneration of sludge acid may be briefly noted in passing. Stolzenwald[3] recovers the sulphuric acid and oils from waste acid by continuously introducing the crude, or water-treated, and heated sludge from below into a vertical heating apparatus of tubular form provided with a dome and gas delivery tube, and heated gradually stronger from the top. The Steaua Romana Petroleum Ges. m. b. H.[4] separates "black acid" in the usual way, and then allows it to flow continuously into pure concentrated sulphuric acid, heated to its boiling point and into which a current of air is passed to oxidize the impurities; the acid vapors are then condensed in a suitable apparatus. The process of the De Bataafsche Petroleum Maatschappij[5] for working the acid tars from refineries consists in diluting the tar with water in such amount that the recovered sulphuric acid has a concentration of at most 52°Bé. The acid tar, so diluted, is then heated to 140° to 165°C. under 7 atmospheres pressure. In the process of the Rütgerswerke A.-G.[6] for

[1] On the preparation of resins from lubricating oil residuum at Baku, see ADIYASIEVITCH, *Zap. imp. russk. techn. obschtsch.*, **29**, 93.

[2] *Chem. Rev. Fett- Harz-Ind.*, **5**, 27.

[3] German Patent 212000, Aug. 6, **1907.**

[4] German Patent 221615, June 19, **1909.**

[5] German Patent 257763, Oct. 2, **1910.**

[6] German Patent 261777, Jan. 12, **1911.**

working up the acid resins from the sulphuric acid washings of mineral oil, the various viscous crude washing acids are mixed together in a funnel-shaped lead vessel, diluted with about one-third part of water and mixed with tar oils, whereupon the mixture is thoroughly agitated at about 70°C. and then allowed to settle. After about 12 hr., the waste sulphuric acid is drawn off as the lower layer, and the separated resin-acid-tar solution is worked further, after the last acid residues have been extracted with soda lye.

**American Practice in the Regeneration of Sludge Acid.**—As early as 1864, R. G. Loftus claimed a process of recovering the acid used in refining Pennsylvania petroleum. According to his patent,[1] the spent acid is, first, diluted with 50 per cent. of water, subjected to agitation and then repose in a lead-lined tank, and the oily matters subsequently drawn off; second, the diluted acid is concentrated by evaporation to from 1.650 to 1.700 and subjected to further dilution and repose; third, the clear liquid is siphoned off from the heavier impurities and again concentrated to from 1.650 to 1.700; and, fourth, it is concentrated in glass, porcelain, or other suitable vessels to a specific gravity of 1.845.

Another early patented process, that of L. S. Fales,[2] relates to effecting the separation of the tarry matters from the spent acid of oil refineries. The spent acid, either with or without the addition of potassium sulphate or of ammonia, and diluted with water, is subjected to the action of ammoniacal vapors from gas liquor, and then allowed to stand, when, it is claimed, the tarry matters are removed, leaving a clear solution, which is then concentrated by evaporation, sodium sulphate being first added.

In the process of A. Penissat,[3] sulphuric acid is recovered from the refuse in the treatment of "coal oil" by washing the acid from the tar, evaporating down to about 60°Bé., and then vaporizing, condensing, and producing the "white" sulphuric acid and concentrating. F. F. Farrar and F. P. Gill[4] reclaim acid from the residuum tar of refineries by mixing the tar with hot water and steeping with heat, then allowing it to cool and settle, when the acid and tar are drawn off from below; the acid water is then heated, the purer liquor withdrawn from the bottom and the water

[1] United States Patent 43157, June 14, 1864.
[2] United States Patent 97182, Nov. 23, 1869.
[3] United States Patent 204244, May 28, 1878.
[4] United States Patent 206309, July 23, 1878.

evaporated. According to the process of H. Bower,[1] sulphuric acid is recovered by washing the sludge acid with water in covered tanks, mechanically separating the sulphuric acid solution and carbonaceous matters from the oily ingredients, as by centrifugal machines, separating the acid solution from the carbonaceous matters by heating in a series of concentrators, and finally concentrating and distilling the separated sulphuric acid solution. E. Clark[2] claimed a procedure wherein the offensive vapors are conducted off by an exhaust produced by an induced steam blast while the sludge is being agitated by steam. R. M. Breinig[3] mixes with the sludge a soap compound adapted to unite with the sludge tar, and then separates the free acid from the tarry mass. H. de Grousilliers[4] recovers sulphuric acid from sludge acid by first removing the petroleum or tarry impurities by floating them, then adding sodium sulphate to the waste acid, precipitating the bisulphate formed by boiling and evaporation, then depriving the precipitate of its aqueous substance by heating to a moderate red heat, and finally vaporizing and condensing the sulphuric acid. W. Waring and J. E. Breckenridge[5] mix about 4 per cent. of sodium nitrate with sludge acid, at a temperature between 60° and 180°F., to purify it and permit the recovery of the sulphuric acid. John L. Gray claims[6] the production of pitch, asphalt, etc., from petroleum sludge by digesting the latter with water, steam or dilute acid recovered from sludge, until the major portion of the acid has been removed, and then heating by a steam spray until the mass is converted into pitch. He also claims a process[7] wherein acid sludge is digested with water, air and steam until the light constituents, including unsaturated hydrocarbons, rise to the top, these then being withdrawn and the digestion continued until a second grade rises, a heavy residuum being withdrawn from the bottom and the weak acid finally separated. In another process of John L. Gray,[8] acid is separated from petroleum sludge by two washings, the weak acid from the

[1] United States Patent 230171, July 20, **1880.**
[2] United States Patent 232685, Sept. 28, **1880.**
[3] United States Patent 306897, Oct. 21, **1884.**
[4] United States Patent 378774, Feb. 28, **1888.**
[5] United States Patent 643578, Feb. 13, **1900.**
[6] United States Patent 923427, June 1, **1909**; see, in this connection, THIELE, *Chem.-Ztg.*, **37** (1913), 841.
[7] United States Patent 923428, June 1, **1909.**
[8] United States Patent 923429, June 1, **1909.**

second washing being used in the first washing of a succeeding body of sludge. Schildhaus and Condrea[1] heat acid tar or sludge to 200°–300°C., and, at the same time, introduce a current of air, heated to about the same temperature, into the retort; the liquid hydrocarbons contained in the distillation products are condensed and the remaining gases, containing sulphurous acid, are washed, first with a heavy hydrocarbon oil, and then with sulphuric acid. Finally, it may be noted that van Tienen[2] recovers hydrocarbons and sulphuric acid from acid tar by mixing therewith sufficient water to dilute the acid yielded by the tar to a specific gravity of not more than 52°Bé., and heating the mixture at a pressure of about 7 atmospheres and a temperature of 140° to 165°C. The liquid is said to separate into two layers, one containing regenerated oils and the other sulphuric acid.

Still more recently some attention has been given to apparatus for recovering refinery acids[3] and to apparatus for concentrating weak acid from sludge.[4]

At the present time there are several American plants which are devoted to the recovery of sulphuric acid from sludge acid; but there are, in addition, a number of smaller plants forming a part of large refineries and not operated independently as in the case of those mentioned.

An acid-recovery plant consisting of the following parts, has been used in some refineries:

1. A separator for removing the tarry matter from the sludge acid. This may consist of a shallow-hooded lead pan, 12 ft. by 55 ft. by 1 ft. deep, with 1,500 ft. of 1-in. tight lead steam coil in the bottom.

2. A series of three shallow lead pans ("weak acid pans"), covered with brick arches. These pans may be 6 ft. wide by 45 ft. long by 1 ft. deep.

3. A series of "strong acid pans," consisting, for the above specified equipment, of two shallow lead pans of the same di-

[1] United States Patent 956184, Apr. 26, 1910. Cf. United States Patent 1010221, Nov. 28, 1911, of J. S. and A. A. Blowski.

[2] United States Patent 1000646, Aug. 15, 1911.

[3] See, for example, Gellen's United States Patent 1063025, May 27, 1913.

[4] In this connection, see Blacher and Sztencel, United States Patent 956276, Apr. 26, 1910; and E. B. Gray, United States Patent 1005425, Oct. 10, 1911.

mensions and two iron stills or pans, 3 ft. by 7 ft. by 9 ft. deep, also housed in brick.

The operation of this plant is as follows: Sludge acid is treated with water to yield a "separated acid" of about 35°Bé., containing about 1.2 per cent. of oil. This separated acid enters the separator and is heated with the steam coil therein to 230°F. Thence it enters the weak acid pans, where heat is either applied to the surface of the acid by means of oil burners or underneath firing is used.[1] At a temperature of 330°F. and a gravity of 56°Bé., it passes thence to the strong acid pans, and from these, at a gravity of 64°Bé., it passes to the iron stills, from which 66°Bé. acid emerges at a temperature of 450°F.

The black, viscous sludge acid from the chemical treatment of the lubricating oils[2] is sometimes combined with the sludge from the burning oils.[3] In any case, the sludge is agitated with water in a lead-lined separator, and the weak acid which is recovered is concentrated first in lead pans and finally in cast-iron stills to a gravity of 66°Bé. The residue is used for fuel or is worked into pitch.[4]

**Treatment with Caustic Soda.**—The caustic soda (sodium hydroxide) which is used after the treatment with sulphuric acid, removes the unchanged petroleum acids and phenols, besides neutralizing and removing the sulpho-acids and the sulphuric acid remaining in the oil. Attempts have been made to replace caustic soda by other reagents, such as sodium carbonate, and the oxides of magnesium, calcium, iron, and aluminum. These have generally been unsuccessful, although milk of lime, or slaked lime, is said to be employed in some small Caucasian refineries,[5] and prominent American refiners state that soda ash is more satisfactory than caustic soda for the treatment of lubricating oils. Michler[6] has recommended the employment of silicate of soda, either alone or in conjunction with caustic soda or soda ash. Processes have also been proposed, but not employed commercially, for dispensing with the alkali treatment

[1] The practice varies. It is the experience of some refiners that a large loss of acid results when heat is applied directly to the surface of the acid by oil burners; they therefore heat the pans by underneath fires.

[2] See p. 463.

[3] See p. 460.

[4] See the process of JOHN L. GRAY, p. 589.

[5] Cf. p. 606.

[6] *Eng. Min. J.*, 66 (1898), 338.

by substituting filtration through powdered glass, sand, clay, fullers' earth, etc., in order to remove the tar products.

**The Recovery of Alkali.**—In 1870, W. Goodaire and G. Stead[1] claimed an improvement in restoring the waste alkali used in American oil refineries. In their process, spent alkali liquor is evaporated to a paste, and then calcined to consume the oleaginous portions, leaving "black ash," which is leached, and the hot filtrated liquid treated with hydrated lime. It would seem that the practice of recovering the alkali used in the treatment of petroleum was begun in the United States shortly after the issuance of this patent, and that the shale oil refiners of Scotland adopted a similar practice in 1874.[2]

Hell and Medinger[3] conducted, in 1874, perhaps the first scientific investigation of the sludge acids and alkali residues, and isolated an acid of the formula $C_{11}H_{20}O_2$. In 1890, Aschan[4] separated from the residues of Baku petroleum hexanaphthenic acid and two acids to which he gave the formulas $C_8H_{14}O_2$ and $C_9H_{16}O_2$. Organic bases corresponding to pyridine bases have also been isolated.

Rosenfeld-Rosian[5] states that the petroleum acids are obtained by treating the alkali residues with the acid residues. The oil which separates out is digested with superheated steam, and then washed with a dilute solution of sodium carbonate. After being heated with water for several hours, it is treated with gaseous hydrochloric acid, and the pure esters of the petroleum acids are obtained.[6] Kharitchkov[7] has considered the commercial values of the principal constituents[8] of alkali sludge.

Guiselin[9] has recommended, especially for Rumanian and similar oils, treatment immediately after distillation with the exact amount of sodium hydroxide solution (35°Bé.) required to

[1] United States Patent 101003, Mar. 22, **1870.**

[2] For Scottish methods, see I. I. REDWOOD'S "Mineral Oils and their By-Products," **1914,** 206.

[3] *Ber.*, **7,** 1216; **10,** 451.

[4] *Idem*, **23,** 867; **24,** 2710; **25,** 3661; *Ann.*, **324,** 1.

[5] *Chem. Tech. Ztg.*, **29,** 177.

[6] For a further account of these acids, see p. 888.

[7] *Tekhn. Westn.*, **1** (1907), 131; *Chem.-Ztg. Repert.*, **31,** 266.

[8] Mainly monobasic naphthene acids of cyclic constitution, according to KHARITCHKOV.

[9] *Mat. grasses*, **5,** 2638.

neutralize the naphthenic acids. He states that no troublesome emulsions are then produced.

Otto[1] has introduced the following method for the recovery of sodium hydroxide from the caustic by-products of refineries: Carbon dioxide is passed through the by-product until the latter is completely saturated. The product then consists of a lower layer, containing in water solution 84 to 90 per cent. of the quantity of sodium hydroxide used, and an upper layer containing naphthene acids and the remainder of the alkali. The lower layer is removed and treated with lime to obtain the sodium hydroxide held in solution.

In the Baku region[2] the alkali which has been used in the refining of petroleum is usually evaporated in cascade to the consistency of soap, calcined and then causticized. The solutions have also been evaporated to a soapy consistency and then used as fuel for the first evaporation of other liquors, as they contain considerable heating power.

Wischin[3] has patented a method for recovering sulphuric acid and sodium hydroxide from Baku refinery wastes, wherein the acid solutions are calcined in cast-iron retorts, with or without the addition of sawdust or coke, and the sulphuric anhydride thus freed is led through a battery of the alkaline solutions, liberating the organic acids and leaving sodium acid sulphate in solution. This solution is then evaporated, and sulphuric anhydride is driven off, leaving sodium sulphate, which is finally worked up into sodium hydroxide and sulphuric acid.

**On Refining Petroleum by Liquid Sulphur Dioxide.**—In the chemical treatment of petroleum sulphuric acid is mainly used for the purpose of removing the resinous matters and certain hydrocarbons such as olefines, these constituents being separated out in the form of residual sludge. For the removal of the aromatic hydrocarbons considerable quantities of sulphuric acid, sometimes in the state of "fuming acid," have to be used, according to the proportion of these hydrocarbons present in the distillate, and this often renders the process costly. The employment of large quantities of sulphuric acid, especially fuming sulphuric acid, has a further drawback: it does not attack only the aro-

[1] *Petrol. Ind. Techn. Rev.*, **2**, 284.

[2] WISCHIN, *Oesterr. Chem. Tech. Ztg.*, **26**, 147. See also MARKOVNIKOV, *Petrol. Ind. Techn. Rev.*, **3**, *Suppl.*, 41 (1900).

[3] *Loc. cit.*

matic and unsaturated hydrocarbons, but also destroys a certain quantity of the saturated constituents; the process has thus the disadvantage of still further reducing the proportion of useful hydrocarbons finally obtained.

In order to avoid the drawbacks of the chemical treatment, it is necessary to substitute a physical method of extraction. The constituents of the petroleum must be treated with a solvent which dissolves the hydrocarbons of the unsaturated and aromatic groups but leaves the saturated hydrocarbons undissolved. In this manner the two classes of hydrocarbons can be separated one from another and employed in the way for which they are suited.

The most important point in the solution of this problem consisted in the discovery of an appropriate solvent, and finally liquefied sulphur dioxide was found to be a satisfactory material.[1] This substance readily dissolves the unsaturated and certain aromatic hydrocarbons, which are responsible for the unsatisfactory burning and odor characteristic of certain kerosenes, and leaves undissolved the useful illuminating oil. Frasch attempted to accomplish this object by the application of alcohol,[2] but liquefied sulphur dioxide is claimed to have certain advantages which alcohol has not. According to Edeleanu, the chief advantage, apart from cheapness, consists in the fact that liquefied sulphur dioxide can be readily recovered from the solution, so that in practice the solvent can be used repeatedly in a cycle of operations, without any appreciable loss.

**The Edeleanu Refining Process with Liquefied Sulphur Dioxide.**—The process of Edeleanu makes use of this peculiar property of liquid sulphur dioxide. If the distillate is agitated with liquid sulphur dioxide at a low temperature, the aromatic compounds are dissolved, but the paraffins and naphthalenes are unaffected. Moreover, owing to the difference in the specific gravity, two distinct layers are formed, so that they may be readily separated from one another.

[1] EDELEANU, *Bull. Am. Inst. Min. Eng.*, **1914**, No. **93**, 2313.

[2] According to United States Patent 951272, FRASCH treats Beaumont or similar petroleum by subjecting at least part of the smoky burning oil fraction to a menstruum, as methyl or ethyl alcohol, which has different actions on the smoking and non-smoking constituents of the fraction, thereby separating a non-smoking kerosene; the menstruum is recovered and used repeatedly. Cf. German Patent 202776, May 18, **1907**, of HERMANN GUTTMANN; and MAZET, *Mat. grasses*, **2**, 1534.

The process is comparatively simple. A certain quantity of the sulphur dioxide is added to the petroleum distillate and is dissolved. This addition is continued until the distillate is saturated, and, from that point, each added portion of sulphur dioxide dissolves the aromatic constituents and separates out with them as a distinct layer. Thus, as the treatment is continued, the distillate becomes poorer and poorer in aromatic hydrocarbons until ultimately a refined distillate is obtained, which contains only a small amount of sulphur dioxide, and is practically free from the objectionable aromatic constituents.[1] However, the nature of this operation depends to some extent on the temperature. With a rising temperature the solubility of the paraffin hydrocarbons and the naphthalenes—which at low temperatures are practically insoluble—increases rapidly; whereas the aromatic hydrocarbons are soluble in sulphur dioxide at all temperatures. It is therefore important, as Edeleanu notes, that the temperature should be maintained at a suitably low level, particularly in the case of distillate rich in aromatic hydrocarbons.[2]

It has been found that the removal of the aromatic hydrocarbons from an illuminating-oil distillate causes not only a diminution of the specific gravity, but also an increase in the burning capacity and illuminating power. The richer a distillate is in aromatic and heavy hydrocarbons, the more prominent is the improvement effected by treatment with sulphur dioxide. The effect of the Edeleanu process is said to be clearly discerned when treating a Bustenari distillate. Whereas the chemical process hitherto employed produces a very inferior illuminating oil which smokes badly, and, when burned in a 14-unit Cosmos

[1] United States Patent 911553; English Patent 11140, May 22, **1908.** See also German Patent 216459, May 23, **1908,** of the DISCONTO-GESELLSCHAFT.

[2] The degree to which the process is affected by the temperature naturally depends on the nature of the material refined. For instance, in the case of a distillate from Borneo, which contained as much as 40 per cent. of aromatic hydrocarbons, treatment with 66 per cent. of liquid sulphur dioxide at −7°C. produced no separation; but a considerable proportion of the aromatic constitutents could be separated at −10°. On the other hand, in the case of a Mexican distillate, which only contained about 17 per cent. of aromatic hydrocarbons, treatment with an equal amount of liquid sulphur dioxide produced separation even at a temperature higher than +10°C. In practice, it is found that each distillate requires special treatment, according to its composition, in order to obtain the best results.

burner, develops only 7 c.p., the treatment with liquid sulphur dioxide produces an oil equal in quality to the best American and Russian oils.

*Utilization of By-products.*—The extract obtained in addition to illuminating oil contains, as mentioned, aromatic hydrocarbons and hydrocarbons rich in carbon. On account of their low burning capacity, these are unsuitable for use as burning oils; nevertheless, by dividing the extract into two products by redistillation, they are said to become useful—the lighter portion as a turpentine substitute or, mixed with light benzine or benzol, as a motor spirit of high efficiency, and the heavier as motor oil for Diesel motors. The extract is, therefore, as regards the possibility of turning it to account, at least equally as valuable as the distillate. Edeleanu[1] states that further experiments have shown that the extract can be resolved by special means into the lower homologues of the aromatic hydrocarbons. By pyrogenic distillation the hydrocarbons contained in the extract are split up, and, in addition to gas, a large percentage of tar is produced. The latter is distinguished from tar derived from coal by its higher content of the lower members of the aromatic series of hydrocarbons.

*Effect of the Edeleanu Process on Distillates Containing Sulphur.*—It is further noted by Edeleanu that liquid sulphur dioxide also possesses the property of dissolving out from crude distillates certain sulphur-containing constituents. For instance, by treating a Mexican distillate of 0.803 specific gravity and with a sulphur content of 0.6 per cent., an oil was obtained which showed only 0.08 per cent. of sulphur; and another oil of 0.79 specific gravity, with a sulphur content of 0.46, was reduced to 0.04 per cent. of sulphur.

*Industrial Operation.*—The Edeleanu process was first tested in a small experimental plant in the Vega refinery at Ploesti, where the results obtained were entirely satisfactory, and shortly afterward the construction of the necessary plant for carrying out the process on a large scale was undertaken. A refining plant was next installed in Rouen, and was followed soon after by the construction of a plant of similar size in Ploesti. The capacity of the plant at Ploesti, originally intended to treat up to 25 tons of distillate per day, was soon increased to 70 tons

[1] *Bull. Am. Inst. Min. Eng.*, **1914**, No. **93**, 2322. This contribution has been freely drawn from in the preparation of the above account.

a day and the nature of the products is claimed to be giving every satisfaction.

These trials were considered to demonstrate completely the practicability of the process on a large scale. A large petroleum concern later ordered a plant with a daily capacity of 250 tons, and in 1914 an increase to 500 tons was contemplated. In 1914, there were other large plants in course of construction in Galicia, Rumania, Borneo, India, etc.

The following description of the treatment of a Rumanian distillate from Bustenari, is given by Edeleanu.[1]

The dried distillate to be treated passes through filters charged with salt, is collected in a receiver, and finally reaches the distillation cooler after going through a cold-exchanging apparatus. The temperature of the distillate is first lowered in the cold-exchanging apparatus and is then further cooled in the distillation cooler itself until it reaches the desired low temperature. Similarly, the liquid sulphur dioxide coming from the vessel passes through a second cold-exchanging apparatus, where its temperature is reduced somewhat, and from thence to the sulphur dioxide cooler itself, where it receives the necessary final cooling. The arrangement is shown in Fig. 201. The distillate cooled to about $-10°C.$ is now let into the lower mixer, a cylindrical vessel which is provided with level gauges, and in its lower part with gauge glasses, so that the reaction in the liquid may be accurately observed.

The introduction of the liquid sulphur dioxide into the mixer begins immediately. The sulphur dioxide is at first entirely absorbed by the distillate, without any appreciable change of the liquid. As soon as the distillate is saturated with the liquid sulphur dioxide, dark cloudy masses begin to rise, and the color changes suddenly to a deep brown. This change of color is brought about by the separation of the liquid sulphur dioxide loaded with the aromatic hydrocarbons. There are now two sharply divided layers which may be distinctly observed through the level gauges. As soon as there is a certain quantity of extract formed, the drawing-off commences; at the same time further quantities of liquid sulphur dioxide are supplied to the mixer.

The extract is drawn by the extract pump through the cold-exchanging apparatus into the extract evaporator. In the cold-

[1] *Loc. cit.*

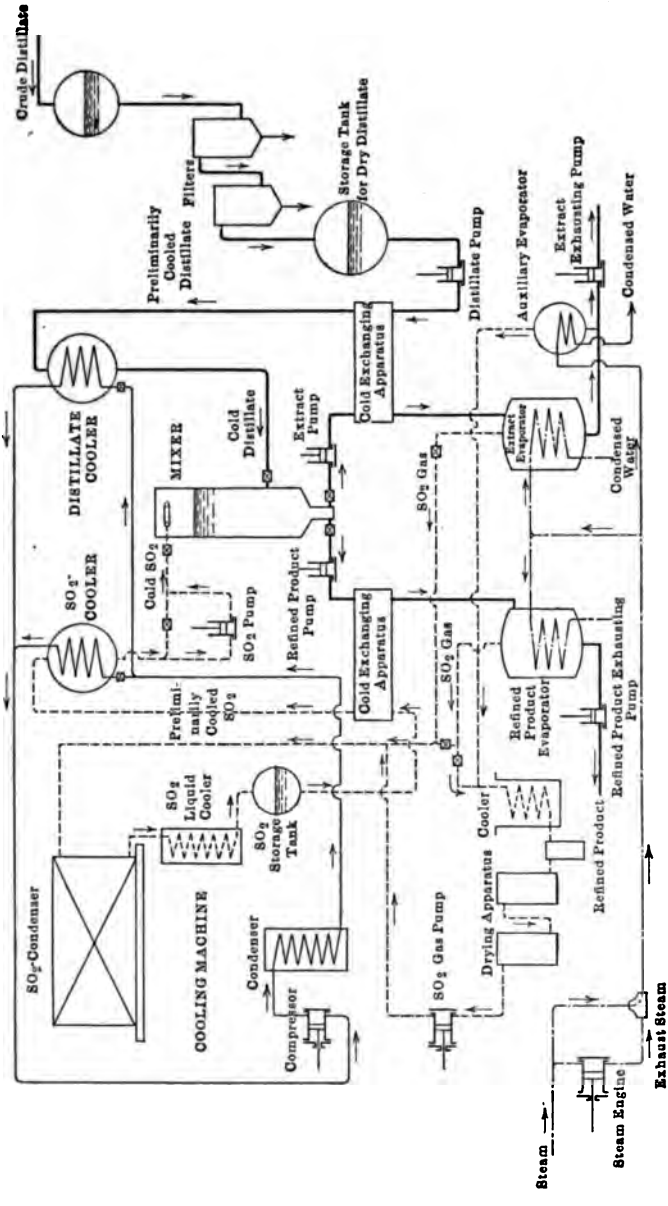



FIG. 201.—Plan of a refinery for the liquefied sulphur dioxide process.

exchanging apparatus it meets the comparatively warm distillate flowing to the distillation cooler (as described above), by which its temperature is considerably raised. In this way the distillate receives a preliminary cooling and the extract a preliminary warming. The latter is then further warmed in the extract evaporator, which is provided with heating coils. This causes the rapid evaporation of the dissolved sulphur dioxide, which flows directly into a condenser, where it accumulates in the form of a liquid; it is then stored in the reservoir, from whence it again commences its cycle.

As soon as the greater part of the sulphur dioxide contained in the extract has been driven off, the evaporation is gradually brought to a stop. The final remaining portion of the sulphur dioxide is held very tenaciously by the extract, and cannot be expelled merely by the application of heat. In order to prepare the extract evaporator for a fresh charge, the extract with its still remaining small content of sulphur dioxide is run into an auxiliary evaporator. Here, the balance of the sulphur dioxide is drawn off by a sulphur dioxide gas pump, under continuous application of heat, and is sent to the condenser. This is continued until about 0.3 per cent. still remains in the extract, when the latter is removed from the auxiliary evaporator.

The refined product is pumped through the second cold-exchanging apparatus into the second evaporator. It meets in the cold-exchanging apparatus the liquid sulphur dioxide flowing from the tank to the cooler, and is raised to practically the same temperature. In this way the liquid sulphur dioxide receives a preliminary cooling (just as in the case of the distillate). The refined product is then collected in the evaporator and undergoes a similar process of evaporation as the extract. By means of heat and continued suction the sulphur dioxide is almost completely removed, only about 0.1 or 0.2 per cent. remaining; at this stage the refined-product evaporator is emptied.

The treated distillate, after being washed and neutralized with an alkaline solution, may be used as finished illuminating oil. In some cases, however, subsequent treatment with very small quantities of sulphur dioxide is said to be advantageous in order to obtain a "water-white" color.

The exhaust steam from the engine which drives the plant, is utilized for the heating of the evaporators.

While the evaporating process is still proceeding in the extract

evaporator, a new operation takes place, a freshly cooled distillate being admitted to the mixer and saturated with liquid sulphur dioxide, until again fresh quantities of extract settle out. By this time the evaporation in the extract evaporator is also finished and the apparatus emptied, so that the new operation may be continued without interruption.

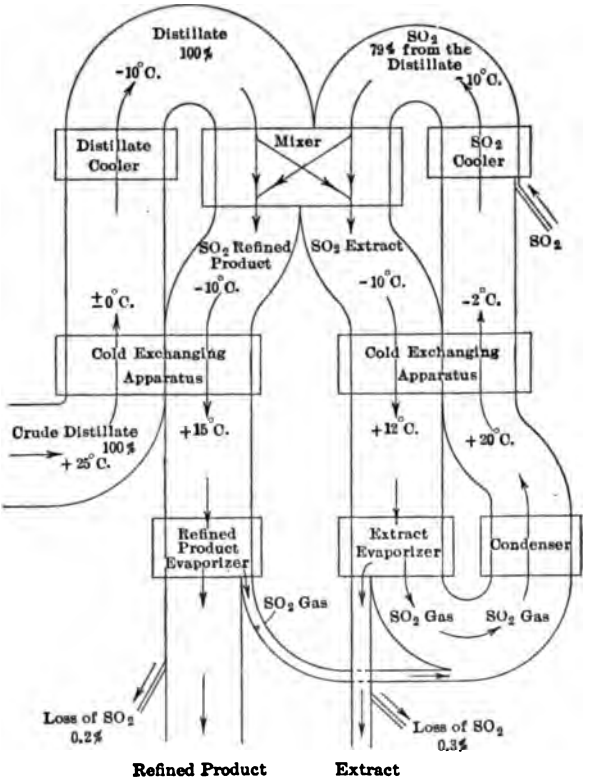



FIG. 202.—Plan of the Edeleanu process.

The cooling of the distillate and sulphur dioxide collected in the two cooling vessels is effected in the usual manner by the aid of cooling coils, which form a part of a separately working cooling machine. The cold-producing medium enters the cooling tubes as a liquid, evaporates on account of the continuous low pressure produced by the suction of the compressor of the cooling machine, and abstracts from its surroundings, *i.e.*, the distillate and liquid sulphur dioxide, the heat required for its evaporation. The

vapor of this cooling machine is afterward sucked up by the compressor and led to the condenser of the cooling machine, where it is again collected in liquid form.

The method of working is shown graphically in Fig. 202. In this diagram it is shown how fresh distillate is continuously entering the system, and how it finally leaves the system separated into refined product and extract, and how the sulphur dioxide, which effects the splitting up, is recovered with the exception of a very small quantity, and is then used again for the process, making a complete cycle. To quote Edeleanu:

"It may also be seen that of the distillate put into the system nothing is lost; the products treated, viz., the volume of extract and the refined product, together are equal in volume to that of the original distillates.

"As the sulphur dioxide performs a complete cycle, it cannot, when once entering the system in a pure and anhydrous condition, give rise to any undesirable complications. It is well known that liquid sulphur dioxide is quite inactive toward metals, so that there is no fear of the apparatus becoming injured or destroyed in the course of time.

"However, great care must be taken that no moisture is introduced into the system with the distillate. The latter should, therefore, as mentioned above, pass through several drying filters before it enters the apparatus, in order that all the moisture may be removed. Inasmuch as the system is perfectly air-tight throughout, there is, of course, no possibility of moisture from the atmosphere penetrating into it.

"The fact that the whole of the apparatus, pipes, valves, etc., are completely air-tight is responsible for the fact that scarcely the faintest odor of sulphur dioxide is perceived in the plant. This is of the greatest importance for the welfare of the workmen who are in attendance at the plant."

The arrangement of the plant is such that from the operator's platform the coolers which are placed high and the evaporators which are below, as well as the mixer in the center, may be watched and attended to. The important pipes with their control valves are placed on a regulating table, so that the operator in charge of the refining process can take care of all the necessary regulating and connecting manipulations without leaving his place of observation.

*Working Results.*—The working results of a plant with an average daily capacity of treating about 65 tons of distillate per 24 hr., as were reached in the plant at Ploesti, are given in the following table, prepared by Edeleanu.[1]

[1] *Loc. cit.*

| | |
|---|---|
| Specific gravity of the crude distillate to be treated | 0.820 |
| Average time of operation, minutes | 68.0 |
| Quantity of distillate treated at each operation, kilograms | 3,115.0 |
| Temperature in the mixer, degrees Centigrade | −10.0 |
| Specific gravity of treated product | 0.8028 |
| Specific gravity of extract | 0.8691 |
| Sulphurous acid left in the treated product, per cent | 0.16 |
| Sulphurous acid left in the extract, per cent | 0.36 |
| Consumption of fresh steam per operation, kilograms | 1,200.0 |
| Consumption of fresh steam per 1,000 kg. distillate, kilograms | 385.0 |
| Consumption of exhaust steam per operation, kilograms | 648.0 |
| Consumption of exhaust steam per 1,000 kg. distillate, cubic meters | 208.0 |
| Cooling water used per operation, cubic meters | 17.1 |
| Cooling water used per 1,000 kg. distillate, cubic meters | 5.5 |
| Loss of sulphurous acid per operation, kilograms | 17.5 |
| Loss of sulphurous acid per 1,000 kg. (distillate), kilograms | 5.6 |

From the above figures, the cost of treating a Rumanian Bustenari distillate can be calculated as follows:

COST OF TREATMENT IN A PLANT OF 65 TONS DAILY CAPACITY

| Cost of Plant: | | Francs |
|---|---|---|
| Machines and apparatus | | 130,000 |
| Buildings, construction, freight, duty and other expenses | | 100,000 |
| Total | | 230,000 |
| Amortization and Interest: | | |
| Amortization | = 10 per cent. of 130,000 | 13,000 |
| Amortization | = 5 per cent. of 100,000 | 5,000 |
| Interest on the invested capital | = 5 per cent. of 230,000 | 11,500 |
| Total | | 29,500 |
| In 325 working days, at 65 tons per day, there will be 21,125 tons treated, so that the amortization and interest amount to fr. 1.396 per ton, or per 100 kg | | 0.140 |
| Maintenance, Labor, etc.: | | |
| Repairs, working materials | | 6,000 |
| Wages and salaries | | 8,000 |
| Insurance, 1½ per cent | | 3,450 |
| Lighting and heating | | 2,400 |
| Drying salt, unforeseen expenses | | 3,000 |
| Total | | 22,850 |
| The cost of maintenance and labor, therefore, amount to fr. 1.082 per ton, or per 100 kg | | 0.108 |

Cost of Materials:

| There are required for each 1,000 kg. of distillate: | Francs |
|---|---|
| 385 kg. fresh steam at fr. 3.50 per ton | 1.35 |
| 208 kg. exhaust steam at fr. 1.50 per ton | 0.31 |
| 5.5 cu.m. cooling water at fr. 0.04 per cu.m | 0.22 |
| 5.6 kg. sulphurous acid at fr. 18.00 per 100 kg | 1.01 |
| Cost of materials per 1,000 kg | 2.89 |
| Cost of materials per 100 kg | 0.289 |
| The total costs of treatment, therefore, amount to | 0.537, |

or practically fr. 0.54 per 100 kg. of distillate, or $0.928 per ton. It must also be taken into account that fr. 18 per 100 kg. of sulphurous acid is a very high figure and refers to imported sulphurous acid. The cost of this material is very much lower when sulphurous acid is used which is produced in the country itself, or actually in the refinery.

COST OF TREATMENT IN A PLANT OF 500 TONS DAILY CAPACITY

| Cost of Plant: | Francs |
|---|---|
| Machines and apparatus | 1,100,000 |
| Buildings, construction, etc. | 500,000 |
| Total | 1,600,000 |

| Amortization and Interest: | | |
|---|---|---|
| Amortization | = 10 per cent. of fr. 1,100,000 | 110,000 |
| Amortization | = 5 per cent. of fr. 500,000 | 25,000 |
| Interest on the invested capital. | = 5 per cent. of fr. 1,600,000 | 80,000 |
| Total | | 215,000 |
| In 325 working days, at 500 tons per day, there will be produced 162,500 tons, so that the amortization and interest amount to fr. 1.323 per ton, or per 100 kg | | 0.132 |

| Maintenance, Labor, etc.: | |
|---|---|
| Repairs, working materials | 40,000 |
| Wages and salaries | 30,000 |
| Insurance, 1½ per cent | 24,000 |
| Lighting and heating | 10,000 |
| Drying salt, unforeseen expenses | 20,000 |
| Total | 124,000 |
| Brought forward | 0.132 |
| The cost of maintenance and labor, therefore, amount to fr. 0.763 per ton, or per 100 kg | 0.076 |

**Cost of Materials:**

| There are required for each 1,000 kg. of distillate: | |
|---|---|
| 385 kg. fresh steam at fr. 3.50 per ton | 1.35 |
| 208 kg. exhaust steam at fr. 1.50 per ton | 0.31 |
| 5.5 cu.m. cooling water at fr. 0.04 per cu.m | 0.22 |
| 5.6 kg. sulphurous acid at fr. 10.00 per 100 kg | 0.56 |
| Cost of materials per 1,000 kg | 2.44 |
| Cost of materials per 100 kg | 0.244 |
| The total cost of treatment | 0.452, |

or, roughly, fr. 0.45 per 100 kg. of distillate, or $0.776 per ton. In this case, the cost of sulphurous acid is taken at fr. 10 per 100 kg., sulphurous acid produced on the spot being reckoned. The cost of production is amply estimated throughout.

The above tables are based on figures said to be actually obtained from the existing plants. It is, however, to be anticipated, that with further perfection of the working of the process, the costs will be reduced even more.

Edeleanu thus summarizes the chief advantages of this process:

1. The process makes it possible to obtain illuminating oil of good quality from the greatest variety of low-grade oils.

2. The sulphur dioxide employed is cheap and practically all of it is recovered.

3. Sulphur dioxide required for the process can even be obtained from the acid sludge available in the refinery in the manufacture of lubricating oils.

Engler and Ubbelohde[1] have investigated the Edeleanu process. Researches on a fraction of Galician petroleum (sp. gr. = 0.8150) showed that the Edeleanu process of refining gave three successive extracts with sp. gr. = 0.9083, 0.8932 and 0.8803, respectively, while the fines amounted to 77 per cent. of the original fraction and had a density of 0.8040. Very similar results were obtained with Mexican and Peruvian oils. When known quantities of unsaturated hydrocarbons (xylene, pseudocumene, etc.) were added to the refined portions, they were found to be almost quantitatively separated by the Edeleanu process. In the commercial operation of the process, the petroleum dis-

[1] *Z. angew. Chem.*, **26**, *Aufsatz.*, No. 27, 177. The EDELEANU purification method has also been considered by GAD, *Petroleum*, **4**, 324; *Oesterr. Chem. Tech. Ztg.*, **26**, 175.

tillate is dried, cooled, and mixed with precooled sulphur dioxide in a tank provided with refrigerating coils, the sulphur dioxide being added in a fine spray. When the desired purification has been attained, the lower layer of liquid (containing the extracted unsaturated compounds) is drawn off and the sulphur dioxide is evaporated off and recovered. The fines are usually pale yellow and may be made water-white by additional refining with 0.5 per cent. of sulphuric acid. They are of lower density than the original distillate and possess a lower density and greater illuminating power than the product refined by the old sulphuric acid process. The extract is yellow to brown in color, and is composed almost entirely of unsaturated hydrocarbons. It is unsuitable for illuminating purposes, but, by virtue of its composition, that portion of the extract boiling below 200° furnishes an excellent turpentine substitute. The higher boiling constituents of the extract are suitable for fuel purposes or for gas production. The cost of the Edeleanu process on the basis of refining 62 tons of crude distillate daily has been found to be 0.436 M. per 100 kg. The process is considered by Engler and Ubbelohde to be a radically new departure and to be of the greatest technical importance.

## OTHER METHODS OF REFINING

It has been proposed to replace sulphuric acid by certain other refining agents,[1] but none of these substitutes has come into general employment. A number of special refining methods follow.

**Treatment with Sulphuric Anhydride.**—As mentioned, oleum, or fuming sulphuric acid, has been used instead of 66°Bé. sulphuric acid, particularly in cases where the oils are difficult to refine; and it has been stated that 5 per cent. of oleum will do the work of 10 per cent. of 66°Bé. sulphuric acid in an air agitator.[2] An objection to its use is that fuming sulphuric acid forms sulphonated products which are left dissolved in the oil treated therewith. These may, however, be removed by agita-

[1] Principally oxidizing agents, as, for example, "chloro-chromic acid," nitric acid, chloride of lime ("bleaching powder"), potassium permanganate, and chromates. The use of chloride of lime was suggested in *J. de l'Eclairage au Gaz*, **1871**, No. 5; for a recent use thereof in refining, see GUILLARD, French Patent 452167, **1912**.

[2] VEITH, *Oesterr. Chem. Tech. Ztg.*, **18**, 7.

tion with a small quantity of 75 to 100 per cent. sulphuric acid previous to the alkali treatment.[1]

John C. Black[2] refines California or similar petroleum by forming outside the oil to be treated a mixture of sulphuric anhydride and a diluent gas,[3] subjecting the petroleum at sludge-producing temperature and in the form of kerosene to the mixture, continuing the treatment until a removal of the smoky or sulphurous hydrocarbons is effected, and separating the sludge from the refined oil. C. J. Robinson[4] refines Beaumont or similar petroleum by subjecting the smoky burning oil fraction to the action of 98 per cent. or stronger sulphuric acid, to remove the smoky hydrocarbons not attacked by 66°Bé. sulphuric acid, and incompletely distilling the treated fraction. Petroff[5] refines petroleum and recovers the sulpho-acids formed by treating the oil with fuming sulphuric acid, allowing the mixture to separate into two layers, washing the upper layer with a mixture of alcohol and water to dissolve the sulpho-acids, extracting the lower layer with gasoline to dissolve the sulpho-acids in it, and washing the solution thus obtained with alcohol and water.

**Treatment with Lime.**—Dorff[6] claims that by treatment of crude oil with lime before distillation, the acidity of the distillate is 25 to 45 per cent. lower than in oil not so treated. The sulphur and unsaturated hydrocarbons are also greatly reduced. By agitation with finely ground lime, Schulz[7] claimed to have obtained as satisfactory refining as by the usual sulphuric acid method. He reported that a large excess of lime and long agitation were necessary. Kharitchkov,[8] however, recommended agitation of the distillate to be refined with milk of lime, then air blowing until cooling was complete, and finally treatment with sulphuric acid and sodium hydroxide. In refining mineral

[1] *Z. angew. Chem.*, **1897**, 629.

[2] United States Patent 968640, Aug. 30, **1910**. This process is owned by the Standard Oil Company.

[3] Such as is obtained by passing the gases produced by burning sulphur in air over catalytic material.

[4] United States Patent 968692, Aug. 30, **1910**.

[5] United States Patent 1087888, Feb. 17, **1914**. On the employment of fuming sulphuric acid in refining petroleum oils, see BOLEG, *Chem. Rev. Fett- Harz-Ind.*, **4**, 263.

[6] *Petrol. Rev.*, **1909**, 187; *Petroleum*, **4**, 928.

[7] *Petroleum*, **5**, 86.

[8] *Naphta*, **9**, 211.

oils, de Fazi[1] distils the oil, after the ordinary treatment with sulphuric acid, in the presence of slaked lime, and then passes the vapors through a mixture of lime and carbon, either in powder form or in blocks.

**Treatment with Sodium.**—Deichler and Lesser[2] have claimed the removal of the sulphurous and other impurities, including aromatic hydrocarbons and phenols, from petroleum or its distillates by treatment with 1 per cent. of sodium. Kharitchkov[3] considers this method capable of economizing caustic soda in refining, but states that treatment with sulphuric acid is still necessary. The advantages are also diminished by the higher consumption of fuel required in the preliminary heating process.

The offensive "cracked" odor of cracked gasoline may be effectively eliminated by treatment with sodium.[4]

**Treatment with Aluminum Hydroxide.**—Gawalowski[5] has found that the fibrous or spongy aluminum hydroxide described by Wislicenus[6] is a very effective agent for the purification of petroleum and benzine. This reagent is said to neutralize any free acid and to exert both a clarifying and deodorizing action. The oil under treatment is agitated for from 4 to 6 hr. with the required amount of reagent and is then filter-pressed.

**Treatment with Various Salts.**—Schwarz[7] states that the use of alkali and alkaline earth sulphates with sulphuric acid, in the purification of petroleum distillates, results in a less energetic action upon the oil, permits of the use of the pitchy residue carried down by the acid, and affords greater efficiency in the recovery of sulphuric acid used in the process.

Gaffart[8] refines petroleum, and especially redistilled oils, by treatment with ferric chloride, strongly acid with hydrochloric acid. Wynne[9] purifies petroleum oils by treating them with a

[1] English Patent 25496, Nov. 15, **1911.** However, as early as 1872, TATRO employed lime instead of caustic soda in refining petroleum (*J. de l'Eclairage au Gaz*, **1872**, No. 3).

[2] German Patent 160717, Jan. 22, **1904.**

[3] *Westnik shirow. promysl.*, **7**, 61; *Chem.-Ztg. Repert.*, **30**, 205.

[4] See p. 579.

[5] *Oesterr. Chem. Tech. Ztg.*, **26** (1908), 87.

[6] *Z. angew. Chem.*, **17** (1904), 801.

[7] *Oesterr. Chem. Tech. Ztg.*, **21**, 161. This process is claimed in SCHWARZ'S German Patent 175453, Mar. 12, **1904.**

[8] French Patent 350091, Aug. 1, **1904.**

[9] French Patent 386651, Jan. 29, **1908**; and English Patent **2314**, Jan. 30, **1907.**

mixture of borax, alum, and sodium chloride, dissolved in boiling water, with the addition subsequently of sulphuric acid, and heating to 23° to 38°C. and passing a current of air to carry off the volatile products. Wohle[1] treats petroleum oils by adding to them an alkaline solution containing salines and a vegetable saponaceous substance, with or without iodides and with or without sulphuric acid. These last three patents describe processes which are of little value on most distillates.

**Miscellaneous Procedures.**—In the process of Richter and Richter,[2] hydrocarbons, such as petroleum, are purified by mixing them with finely divided carbon having catalytic properties, and then bringing the mixture into intimate contact with oxygen, ozone, or gases containing oxygen. It is claimed that impurities, especially those due to the presence of sulphur and nitrogen, are oxidized and carried away in the form of gases. The process of Knottenbelt[3] consists in treating petroleum oils by separating by distillation fractions suitable after chemical refining as substitutes for turpentine and for illuminating purposes, respectively. This treatment consists, in the case of the lighter fraction, in acting on the same with nitric oxide, and in the case of the heavier fraction (after any tarry substances have been separated therefrom) with a solution of ammonia carrying litmus. The refining value of such substances is of little value. Melamid and Grötzinger[4] have claimed a method of purifying oils by means of phosphoric acid or a mixture of this acid with pyrophosphoric and phosphorous acids: a mixture of 1,000 kg. of petroleum with 100 kg. of phosphoric acid, or with 65 kg. of this acid, 10 kg. of pyrophosphoric acid, and 2 kg. of phosphorous acid, is heated to 350° to 400°C. and the "purified" oils are distilled off. Ellis[5] treats heavy petroleum oils containing gum-forming ingredients, to render them suitable for use as lubricants, by exposing them in thin layers to ultraviolet light of great intensity, to debloom the oil and polymerize the gum-forming constituents.

[1] English Patent 23348, Oct. 12, **1909.**

[2] English Patent 23467, Oct. 10, **1910.**

[3] English Patent 12001, May 21, **1909.** On this process, see *Petrol. Rev.*, **30,** 69; *Petroleum*, **6,** 196.

[4] French Patent 443650, May 10, **1912**; addition, Sept. 30, **1912.**

[5] United States Patent 1089359, Mar. 3, **1914.**

## THE DESULPHURIZATION OF MINERAL OILS[1]

It is a well-known fact that the impurities contained in crude petroleum influence the price to a remarkable degree. The best illustration of this is the case of Ohio oil. This contains about 0.75 per cent. of sulphur, and, prior to the introduction of the Frasch desulphurizing process,[2] was sold at 14 cts. per barrel, while Pennsylvania oil, with a sulphur content of only 0.03 per cent., sold at $2.25 at the same time.

One of the petroleums which contains an objectionable amount of sulphur is that found near Petrolia, Ontario. When this crude oil was discovered in 1868, it was refined in the customary manner, namely, treated with sulphuric acid and sodium hydroxide; but the odor of the products was very offensive, indeed, so much so that it has been said that cargoes of ships carrying flour and bacon, anchored near a vessel loaded with Canadian oil, were spoiled, since the flour and bacon absorbed this odor. Law suits based upon these facts were decided against the shippers of Canadian oil and all exports ceased. In order to protect the home industry, the Canadian government imposed a duty of 9 cts. per gallon on Pennsylvania petroleum, but, notwithstanding this prohibitive duty, one-half the oil consumed in Canada was imported from Pennsylvania.

It was only to be expected, therefore, that the Canadian government as well as the producers and refiners of petroleum in Canada, exerted every effort to discover a method by which the objectionable properties of Ontario petroleum could be eliminated. Practically nothing was accomplished for 20 years, but

[1] On the sulphur content of petroleums, see ENGLER, *Chem.-Ztg.*, **20** (1896), 197; on the sulphur compounds in petroleum, see KAST and LAGAI, *Dingler's polyt. J.*, **284** (1892), 69; on sulphur in petroleum, see LOHMANN, *Chem.-Ztg.*, **35** (1911), 1119; for a consideration of the sulphur compounds in Canadian petroleum, consult MABERY and QUAYLE, *J. Soc. Chem. Ind.*, **19** (1900), 505, and *Proc. Am. Acad.*, **41** (1905), 87; and on the sulphur compounds in Ohio petroleum, see MABERY and SMITH, *Am. Chem. J.*, **13** (1891), 233, and **16** (1894), 83. MABERY and SMITH have also discussed the sulphur compounds in crude petroleum and its products in *Ber.*, **22** (1889), 3303. On sulphur compounds in "cracked" gasoline, see p. 579.

[2] The account of the Frasch process which is presented above, has been largely taken from an address by HERMAN FRASCH, published in *J. Ind. Eng. Chem.*, **4** (1912), 134. This process has also been described by LUNGE in *Z. angew. Chem.*, **7** (1894), 69; and by RICHE and ROUME in *Ann. Mines*, (9), **5** (1894), 95.

in 1885 the late Herman Frasch bought a refinery in Canada and began a thorough investigation of the character of this crude petroleum. Frasch found that the penetrating odor, referred to by Canadians as "skunk," was produced by the presence of a peculiar hydrocarbon-sulphur compound; and that when the sulphur was eliminated completely from the oil, the latter furnished an illuminant equal in quality to the best Pennsylvania kerosene. He found that the removal of elementary sulphur or hydrogen sulphide from petroleum presented comparatively little difficulty; but that the sulphur compound which was the cause of this offensive odor, was very stable and could not readily be broken up into any simpler sulphur compound which could be eliminated. It was finally learned, however, that this sulphur compound possesses the property of dissolving a number of metallic oxides; that when the oil is saturated with all the oxide it can carry in solution, the disagreeable odor disappears; and that this solution of metal in petroleum has an intense affinity for sulphur. Frasch finally selected copper as the most suitable metal. A 1200-bbl. still was erected for the purpose of conducting the process on a large scale, and, in this manner, Frasch produced a burning oil containing 0.02 per cent. of sulphur.

About this time, petroleum almost identical with the Canadian product was discovered in Ohio, and while the question of refining Canadian oil was interesting, the problem became intensely important when 30,000 bbl. per day were being produced in Ohio. The new field proved to be very extensive, and the Standard Oil Company bought property near Lima, Ohio, upon which they erected a large refinery. This company disregarded the great difference in the sulphur content of the petroleums of Pennsylvania and Ohio, and decided to refine the latter in the usual manner; but when the refined product had been distributed among their customers, it was all returned as unfit for use. Every effort was made to solve this problem, but in 1887, after two years of experimentation, it was decided that illuminating oil could not be made from Lima petroleum and that this was fit only for fuel. Accordingly, a pipe line for this purpose was built to Chicago and long term contracts were made at 14 cts. per barrel.

At this time, patents had been granted to Frasch, who was selling refined Canadian oil with a guarantee that it would burn equal to the best Pennsylvania product. After an investigation, the Standard Oil Company purchased the patents and refinery

of Frasch, located at London, Ontario, and also proceeded to construct large works for carrying out Frasch's inventions at Lima, Cleveland, Whiting, Olean, Philadelphia and Bayonne. Subsequently, the Ohio field was found to extend into Indiana and the production increased to 90,000 bbl. per day; the price of this crude oil went up to nearly $1.00 per barrel, fluctuating between 60 cts. and $1.00 for a number of years.

**The Frasch Method.**—In desulphurizing petroleum according to the process of Herman Frasch,[1] the cupric oxide is mixed with the petroleum distillate in a still equipped with flyshaft and arms radiating therefrom, to maintain the oxide in suspension during the process of distillation; flexible chains are also attached to the arms to prevent the oxide from adhering to the bottom of the still. 5,000 lb. of cupric oxide are used with the first charge of 1,000 bbl. of distillate. After 80 per cent. has been distilled off, a new charge of distillate is added with an additional charge of 5,500 lb. of oxide; this is followed by three further runs with 5,500, 5,500 and 7,500 lb. of oxide, respectively, so that 29,000 lb. of cupric oxide are used for 5,000 bbl. of distillate. The residuum is pumped through a filter press, and the solid product of the filter press is burned free from oil. The dry mixture of oxide and sulphide of copper is then put into a roasting furnace, where it is desulphurized to 1 per cent. or less.

A variation of the Frasch method treats the oil vapor coming from a still charged with crude petroleum, and consists in passing the vapor through two brushes made of No. 10 steel wire, the brushes being 5½ ft. in diameter and 16 ft. long. They are inserted into a shell which is almost the same diameter as the brush, and the requisite amounts of cupric oxide and heavy oil are pumped into two shells, each containing one brush. The vapor passes in series, first around the shell to prevent condensation; then through the shell on to the condenser. The brushes are made to revolve at about 6 revolutions per minute. These wires immersing continually in the oxide of copper mixture, the shells containing the brushes are surrounded by the vapor coming from the still; and as the temperatures of the vapors increase, anything condensed thereon during the prior period is re-evaporated by the hotter vapor following. In this way the whole of

[1] According to RICHE and ROUME (*loc. cit.*), this process is similar in principle to that adopted in France for desulphurizing glycerin in the soap manufactories at Marseilles.

the volatile constitutents of the oil are subjected to the desulphurizing treatment.

Both of these processes are now in operation in this country.

**Other Desulphurizing Processes.**[1]—Frasch[2] has also proposed distilling the oil over lead oxide, then refining with sulphuric acid; Kennedy[3] has claimed a process wherein the oil is treated with a solution of copper sulphate, sodium hydroxide and sodium chloride, this being followed by digestion with sulphur; Pitt and Van Beck[4] have proposed passing the oil vapors from the still over heated iron or copper; Gordon[5] distilled the oil over lead oxide and washed the distillate with magnesium sulphate, the purification being completed by the usual treatment with acid and alkali; Friedel and Crafts[6] suggested that the sulphur be removed by treatment with aluminum chloride; and dilute nitric acid has been used in the place of sulphuric acid for treating Canadian oils.[7]

In the process of Adiassewich,[8] mineral oils are heated under pressure successively with dilute sulphuric acid, caustic soda, and aluminum chloride. For example, the burning oil obtained by the distillation of shale oil is heated, with about 0.75 per cent. of its weight of 60 per cent. sulphuric acid, to 40°–50°C., under a pressure of 15 to 20 lb. per square inch, for about half an hour, the whole being agitated during the process. The oil is separated, washed, and treated with about 0.3 per cent. of its weight of caustic-soda liquor (specific gravity, about 1.32) at a temperature near the boiling point of the oil, the pressure being maintained at 25 to 30 lb. per square inch. This treatment is continued until the mixture is completely emulsified. The oil is then washed, dried, and heated with about 2 per cent. of aluminum chloride to about 150°C. under a reflux condenser. The pressure is then raised to 10–12 lb. per square inch and maintained for about 3 hr., when the oil is separated, washed with water containing a little lime, and finally distilled. The temperatures and pres-

[1] These processes are included to show the trend of chemical investigation; most of them are probably of no commercial value.

[2] United States Patent 378246, **1888.**

[3] German Patent 43145.

[4] German Patent 45958.

[5] United States Patent 451724, **1891.**

[6] *Compt. rend.*, **84** (1878), 1392.

[7] Tumski's "Technology of Naphtha," Moscow, **1891** (Russian).

[8] English Patent 4431, **1903.**

sures used in general vary with the boiling point of the oil to be treated.

The Macalpine process of refining, which has been recommended as a desirable substitute for the acid and alkali treatment, consists in first subjecting the crude oil to a "salining" operation, whereby it is brought into intimate contact with a saline solution, preferably of a mixture of sodium carbonate and chloride; adding to the separated oil from ⅛ to 1½ per cent. of a manganese compound produced by the decomposition of acetylene by means of permanganates of the alkalies; and then effecting the fractional distillation of the oil under a vacuum of about 700 mm., with the introduction of steam during the later stages. It is claimed that by this process a larger yield of products of improved quality is obtained. Engler, as the result of an experimental investigation, supports the statement of the inventor that by the combined effect of the "salining" and of the oxidizing action of the manganese compound, resinous and asphalt-like constitutents, petroleum-acids, and sulphur are eliminated, illuminating and lubricating oils being thus obtained which compare favorably in purity with those yielded by the ordinary system of refining. Dupré and Redwood found that in the treatment of a Texas crude petroleum by the Macalpine process the percentage of sulphur was reduced from 1.35 to 0.5 per cent.[1]

Hellsing[2] deodorizes and desulphurates mineral oils by treatment with one or more solutions of salts of heavy metals, if desired, in the presence of ammonium chloride and alkalies; Robinson[3] desulphurizes Lima oil by treating a distillate lighter than 0.865 specific gravity with sulphuric acid stronger than 66°Bé. at an initial temperature below 14°, not more than 60 lb. of acid to the barrel being used; Walker[4] desulphurizes petroleum distillates by passing them in a state of vapor over anhydrous copper chloride and then through an alkaline lead solution, as sodium plumbate; and Sherman[5] causes the vapor to pass through a deep layer of granular filling, chemically inert thereto and offering such resistance to the passage of the vapor as to raise the

[1] Redwood's "A Treatise on Petroleum," 3rd. ed., **2**, 30.

[2] English Patent 9180, Apr. 19, **1907**; French Patent 377197, Apr. 26, **1907.**

[3] United States Patent 910584, Jan. 26, **1909.** This is owned by the Standard Oil Company.

[4] United States Patent 955372, Apr. 19, **1910.**

[5] United States Patent 968088, Aug. 23, **1910.**

vapor pressure within the still and the temperature of the issuing vapor above that due to the normal boiling point of the oil and sufficient to eliminate grease and sulphur and cause the deposition of carbon on the inert filling. The process of John C. Black[1] has been referred to.[2]

Finally it may be mentioned that a Wyoming clay containing a considerable amount of magnesium,[3] has been employed in the purification of petroleum containing a high percentage of sulphur.[4] The oil, preferably a distillate, is treated first with a solution of lead acetate, to remove as much of the sulphur as possible. One pound of the Wyoming clay is then added to every 50 gal. of the treated oil; after settling, the oil is decanted, treated with concentrated sulphuric acid, and clay is again added. After these treatments, the oil is agitated with an alkaline solution until it shows no blue coloration with a solution of sodium plumbate

### THE DECOLORIZATION OF PETROLEUM DISTILLATES[5]

By far the greater part of the fullers' earth produced in the United States is employed in clarifying mineral oils.[6] It is mined for this purpose at Ellenton, Quincy and Midway, Fla.; at Bakersfield and Vacaville, Cal.; and in Alabama and Georgia.

A common practice in decolorizing petroleum products is to dry the earth carefully, after which it is ground to suitable sizes and run into vertical cylinders, usually about 12 ft. in length, through which the oils are allowed to percolate slowly until the color reaches a certain maximum shade (see Fig. 203).[7]

A procedure which has been used with success is along the following lines:

Dehydration of the earth is necessary. This operation is con-

[1] United States Patent 968640, Aug. 30, **1910.** This patent is owned by the Standard Oil Company.

[2] See p. 606.

[3] The composition has been given as follows: Silica, 63.25; aluminum, 12.62; ferric oxide, 3.75; magnesium, 3.98; potassium and sodium, 8.16; sulphur, 1.58; and water, 6.71 per cent.

[4] *Chem. Tech. Ztg.*, **30,** 125.

[5] On the employment of bone-black in decolorizing paraffin, see p. 767. On the use of ashes of rice hulls in decolorizing oils, see LASHER, United States Patent 1075481, Oct. 14, **1913.**

[6] See PARSONS, *Bureau of Mines Bull.* **71** (1913).

[7] On the advantages of and objections to this filtration method, see *J. Soc. Chem. Ind.*, **23,** 755.

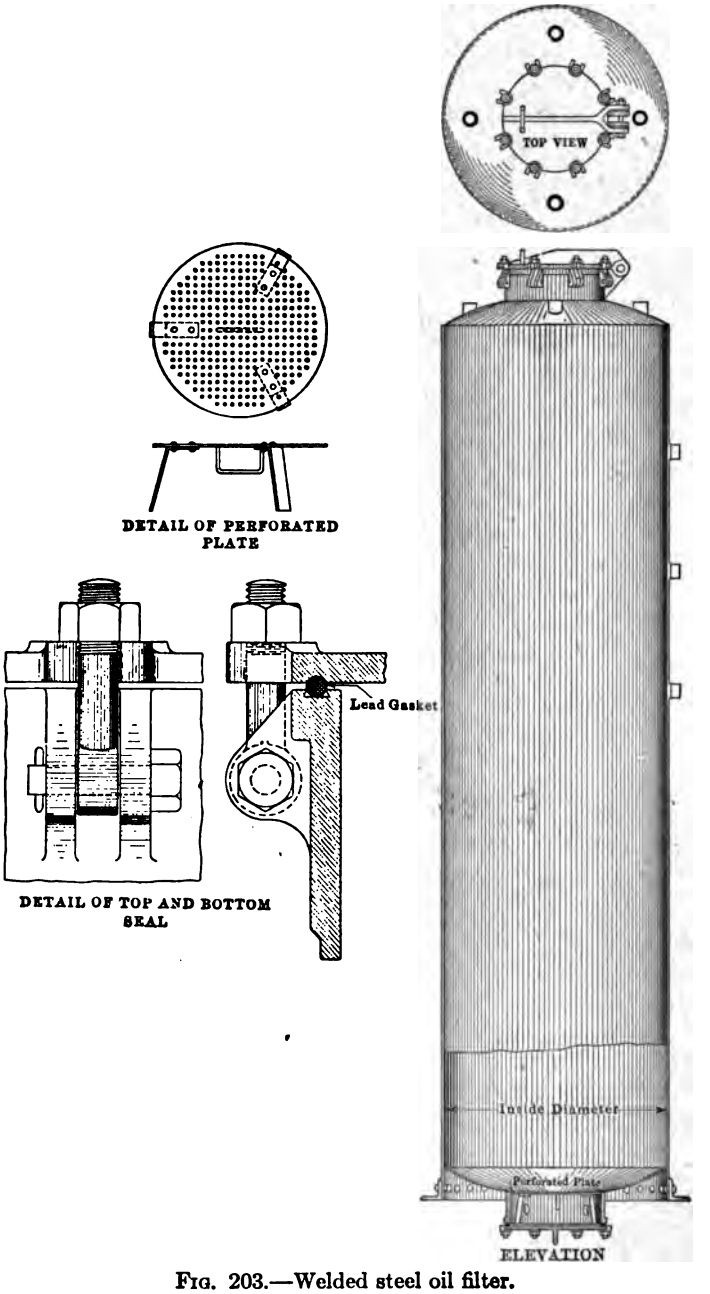



FIG. 203.—Welded steel oil filter.

ducted in a cylindrical, internally fired[1] rotating dryer, from which air is excluded as far as possible, the powder being heated with continual agitation at 300° to 500°C., until it assumes a grayish blue shade. This eliminates both the absorbed water and the chemically combined water of hydration. For many purposes it is sufficient to expel absorbed water by heating the powder at 120°C., though the product is said to be a much less effective bleaching agent than that roasted at the higher tem-



FIG. 204.—An oil filter, 6 ft. by 8 ft.

perature. Preliminary experiments should be made with the oils to be bleached, in order to determine the most suitable temperature in each case. Thus, light mineral oils, such as kerosenes and vaseline oil, are completely decolorized at 12°–17°C. In the case of the heaviest mineral oils, such as cylinder oil, bleaching can only be effected at higher temperatures. The amount of earth required also varies with the different oils,

[1] Oil or gas is used as the fuel.

some requiring 3 to 5 per cent., others 7 to 10 per cent., while others again need as much as 15 to 20 per cent. or more. Hard paraffin, after preliminary treatment with sulphuric acid, etc., is completely bleached by means of only $1\frac{1}{2}$ to 2 per cent. of earth. Finally, the petroleum product, brought to the right temperature, is mechanically mixed for about 20 min. with the requisite proportion of earth, and then pressed in a filter press. The earth left in this process contains about 80 per cent. of its weight of oil. The residual earth can be regenerated by heating it to 400°–500°C., and the dark gray to black product thus obtained can be used again for bleaching purposes, preferably after the addition of 15 to 20 per cent. of fresh earth.[1] It should be borne in mind that certain samples of fullers' earth show liability to spontaneous combustion when soaked with oil, and accordingly great care must be exercised when operating with these.

Guiselin and Haudricourt[2] state that argillous earth must be perfectly dried to bring out its highest decolorizing properties; that damp earth will absorb oil without affecting it in any way; and that the earth should be granular if the filtration is to be through it, but that if it is to be agitated with the oil, it should be powdered. Gräfe,[3] who experimented with fullers' earth from Fraustadt in Silesia, found that this earth gave more satisfactory results after being dried and that it acted best after it had been heated to 400°C., when the water of constitution was expelled. Wesson[4] has reported that if the water of constitution is driven off completely, the fullers' earth becomes useless for bleaching vegetable oils, though perhaps not for mineral oils. Parsons states[5] that water of composition is not an essential factor in the bleaching properties of all fullers' earths, for some bleach fully as well after it has been driven off as before and others lose much of their bleaching power when this water is removed.

[1] In order to prepare it for decolorizing mineral oils, M. J. Welsh (United States Patent 1159450, November 9, 1915) treats raw fullers' earth with hot hydrochloric acid, to dissolve alkaline earth compounds and other impurities tending to fuse or sinter during calcining, and then washes and calcines the product.

[2] *Petrol. Rev.*, **27**, 151.

[3] *Petroleum*, **3** (1907) 292.

[4] *Min. Eng. World*, **37**, 667.

[5] *Bureau of Mines Bull.* **71**, 6.

Gräfe[1] regards the decolorization of petroleum products by fullers' earth as a physical and not a chemical process, and Wesson[2] has observed that the bleaching property seems to depend upon the physical condition of the earth and is closely allied to the proper amount of hydration of the complex silicate; but Löb[3] has objected to the view that the bleaching of oils by silicates is a mechanical phenomenon and considers it a chemical reaction, due to the supposed presence of some substance in the silicates which combines with the coloring matter of the oils and thereby renders it insoluble.[4] Of interest in this connection is

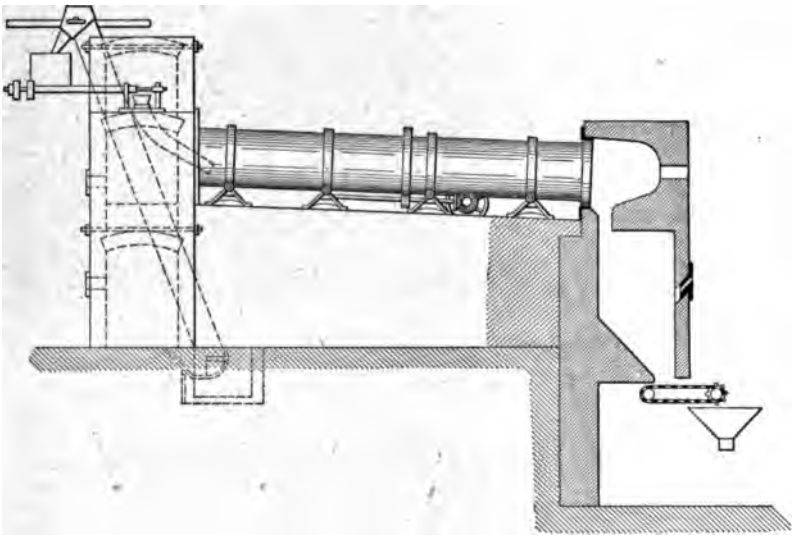


FIG. 205.—The White-Fulton furnace, which has been used in regenerating Florida earth.

[1] *Petroleum*, **3** (1907), 292. A similar view is held by GUISELIN and HAUDRICOURT, *Mat. grasses*, **5**, 2815, 2845, 2882.

[2] *Min. Eng. World*, **37**, 667.

[3] *Chem. Rev. Fett- Harz-Ind.*, **15**, 80.

[4] LÖB cites experiments which seem to support this view. He reports a series of comparative bleach tests which he carried out with scale paraffin, using 5 per cent. of bleaching agent and heating the paraffin with same for 15 min. to 110–50°C. The results are given in the order of their superiority: (1) Bavarian fullers' earth, not ignited; (2) prussiate of potash residues; (3) Florida fullers' earth, not ignited; (4) Florida fullers' earth, ignited; (5) Bavarian fullers' earth, ignited; (6) Silesian fullers' earth, not ignited; (7) Silesian fullers' earth, ignited. The united filtrates of these tests were treated a second time with the same results, except that the prussiate of potash residues take fourth place instead of second place.

the opinion of Porter,[1] that the clarifying action of fullers' earth is due chiefly to the presence of colloidal silica. In order to explain the phenomena observed by diffusing California petroleum through fullers' earth, Gilpin and Schneeberger[2] regard petroleum as an emulsoid and fullers' earth as a dialyzing septum, permitting the free passage of the paraffin oils and causing the adsorption and coagulation of the bitumens, and carrying with it the sulphur and nitrogen compounds and the benzene and olefine hydrocarbons. Gurwitsch[3] maintains that, during the decolorization process, Florida earth effects a polymerization of the unsaturated compounds of the oil treated.



FIG. 206.—Rotary clay burner, 6 ft. by 60 ft., used in burning Florida earth in paraffin wax plants. A rotary cooler is located underneath the floor.

Mineral oils may also be decolorized by filtration through finely-ground, ignited bauxite;[4] by agitation with ground cal-

[1] *Bull.* **315**, *U. S. Geol. Survey*, **1907**, 268.

[2] *Am. Chem. J.*, **50**, 59.

[3] *J. Russ. Phys.-Chem. Soc.*, **47** (1915), 827. GURWITSCH states that when Florida earth which has been used for decolorizing petroleum oils is extracted with ethyl ether, the latter distilled off and the residue mixed with the original oil, the product has a much darker color than it possessed before the treatment with the earth. GURWITSCH ascribes this to polymerization by the earth of the unsaturated compounds of the oil.

[4] GAUTIER, *Chimiste*, **2**, 99; see also note in *J. Ind. Eng. Chem.*, **7** (1915), 353.

careous sandstone;[1] and by passing them in admixture with hydrogen over powdered nickel or other metal, heated to 100 to 350°C., the hydrocarbons having previously been conducted through a column of copper heated to above 350°C.[2] Ubbelohde and St. Philippide[3] have investigated the effect of fullers' earth, unburned kaolin, ignited alumina, and nickel upon petroleum at temperatures varying from 200° to 450°C.; they found that the evolution of gas increased with the rise in temperature, and that the decomposition temperature with nickel was 300°C., with kaolin 250°C., and with fullers' earth 200°C.

## THE PRODUCTION OF ASPHALT BY THE OXIDATION OF PETROLEUM

In 1865, Gesner stated[4] that "organic substances are oxidated by the atmosphere, and its action promoted by a high temperature. *Hot air has therefore been forced through hydrocarbon oil during the process of purification*, and, in some instances, with advantage." However, Jenney[5] was the first to report on the formation of solid oxidized hydrocarbons resembling natural asphalts by the action of air on petroleum.

In conducting a series of experiments on the oxidation of petroleum by heating it to a temperature slightly below its boiling point, and passing a current of oxygen slowly through it, the action being sometimes assisted by the addition of litharge or potassium permanganate, Jenney noticed that a solid deposit formed. It would appear from his work that the more volatile hydrocarbons were distilled away and that oxidation and distillation proceeded simultaneously. In the first experiment conducted by Jenney, air was rapidly aspirated through a crude Pennsylvania petroleum. The temperature was kept at from 140° to 145°C. However, the experiment was not continued long enough to produce oxidized solid hydrocarbons. In the second experiment reported by Jenney, a petroleum distillate of 41°Bé. was used. This was maintained at about 110°C. for

[1] TAYLOR, English Patent 20,409, Sept. 1, **1910.**

[2] HALLER, SABATIER and SENDERENS, French Patent 376490, June 14, **1906.**

[3] *Petroleum*, **7**, 1233.

[4] "A Practical Treatise on Coal, Petroleum and Other Distilled Oils," 2nd. ed., **1865**, 128.

[5] *Am. Chemist*, **5** (1875), 359.

2 days and a rapid current of air was passed through it. The oil was but slightly affected. However, on increasing the temperature to 140°C. and adding small quantities of litharge to increase the action of the air, and continuing 3 days longer, an asphaltic product was obtained. In the third experiment a lubricating oil of 28°Bé. was taken. This was subjected to a temperature of 140° to 155°C., and to the action of litharge and air for 4 days. The product obtained resembled grahamite. Jenney stated that with oxygen at 270°C., "hydrogen was removed from the oil in the form of water by the action of the air."

Jenney obtained patents[1] for the preparation of a substance similar to resin from the "sludge oil" of petroleum refining. According to his claims, the sulphuric acid which has been used for refining petroleum or shale oil, is mixed with an equal volume of water, when two layers form; the upper consists of a thick oil of objectionable odor, the lower of dilute sulphuric acid. The oil is repeatedly washed with boiling water, finally with the addition of a little soda, and is then distilled. After the oil distilling below 250°C. has come over, a current of air is blown by means of a suitable arrangement for 48 hr. through the residual oil, the operation being conducted in the retort used for the distillation. During this process the oxygen of the air is said to be absorbed by the oil, which, on cooling, solidifies to a deep brown mass similar to resin.

Mullerus reported[2] in 1892 that he had heated mineral oil to 120°–150°C. and passed air through it. After a short time, emulsification with sodium hydroxide could be effected. Baku petroleum "wurde durch Einleiten von luft dunkelbraun, trüb und merklich consistenter; das dunkelbraun und trübwerden rührt höchst wahrscheinlich von der *Bildung asphaltartiger Substanzen* her. Wird das oxydirte Oel mit Natronlauge gekocht, so scheiden sich asphaltartige schwarze Flocken aus, ein Beweis, dass sick *durch die Oxydation pechartige Producte gebildet* haben." No formation of asphaltic substance was observed by Mullerus upon treatment of Pechelbronn oil.

As the result of an investigation on the "Influence of Sunlight and Air on Petroleum Products; Capacity of Petroleum for

[1] German Patent 3577; *Dingler's polyt. J.*, **232** (1879), 285; United States Patent 178061 of May 30, **1876.**

[2] *Chem.-Ztg.*, **1892,** No. 76, 1396.

Absorbing Atmospheric Constituents," R. A. Ostrejko[1] stated that on exposure to sunlight and air, ordinary Baku petroleum of medium quality deposited a yellow sediment and became strongly acid, giving off at the same time a penetrating odor. Part of the deposit was soluble in distilled water, a dark tarry substance being obtained on evaporation, and the remainder was dissolved by ethyl alcohol and concentrated sulphuric acid. Several samples of oil exposed in clear glass bottles to air and sunlight became opalescent in 9 hr., and subsequently turbid, with formation of sediment; whereas when kept under the same conditions in green bottles, the oils became appreciably lighter in color. Further tests revealed the fact that in vessels of clear and blue glass, with admission of air, the oil darkened considerably on exposure to light; while in orange-yellow and green bottles the color became lighter in 48 hr.; and in the case of clear glass, with exclusion of air, scarcely any change occurred. The percentage of acidity increased some fivefold in 3 days in the blue and clear glass vessels, but remained practically unaltered in the others. Several distillates refined in different ways all showed that the amount of acidity resulting on exposure increased concurrently with the deepening of the color.

According to Ostrejko, the effect of light in facilitating the absorption of air by the oil is very marked, a sample in a tube, into which a single bubble of air was admitted, absorbing it completely; whereas in a second sample, similarly treated, but kept in the dark, no absorption occurred, the air bubble, on the contrary, appearing to have increased in size.[2]

The experience of Ostrejko[3] with regard to the acid process in

[1] *Trudi bak. otd. imp. russk. techn. obschtsch.*, 1896, **10**, (2), 21.

[2] For the purpose of determining whether the degree of refining influenced the absorptive capacity of the oil toward air, a number of vessels were filled with ordinary oil, and others with oil that had been exposed for some time to light and undergone a subsequent refining with excess of acid and alkali. To the vessels were affixed eudiometer tubes containing 5 c.c. of air, and the whole was exposed for 34 days, under various conditions. The alterations in the volume of air were noted daily, with the following results: Ordinary distillate under the influence of light took up all the air in 10 days; distillate refined with excess of soda required 11 days; ordinary petroleum, 12 days; oil refined with excess of acid and alkali, 34 days. The oil treated with excess of acid only, absorbed but two-thirds of the volume of air during the full period, and the sample previously exposed to light and afterward refined over again had taken up barely any.

[3] *Trudi bak. otd. imp. russk. techn. obschtsch.*, **10**, (4), 19.

refining led him to state that oil refined in this manner has a more agreeable odor, and is less inclined to become cloudy, absorb air, and oxidize, than when the alkali method alone is practised.[1]

In the continuation of his researches, Ostrejko[2] found that crude solar oil distillate under the same conditions, absorbed the air in the eudiometer tube in 12 days, and deepened in color from 3 to 2 mm. on the colorimeter scale in 40 days, whereas refined distillates required in one case 14 and in another 41 days for the absorption of the air, the color changing from 33 to 7 mm. and from 58 to 6½ mm., respectively; the acid was about double. The experiments were also extended to the absorptive capacity of petroleum for carbon dioxide, nitrogen, oxygen, hydrogen, and nitrogen monoxide. At the end of 9 hr. exposure in the dark, nitrogen monoxide, carbon dioxide, and oxygen were found to have decreased in volume, but the converse was noticed with nitrogen and hydrogen, and the same results were obtained by a further 32½ hr. exposure in the dark. On the admission of light, absorption proceeded in all cases, the following times being required for completion: nitrous oxide, 3¾ hr.; carbon dioxide, 4 hr.; oxygen, 8 hr.; nitrogen, 2 days (1 hr. longer than air); hydrogen, 4 days. According to Ostrejko, *this faculty of petroleum for absorbing gases may exert an influence on its composition*; *e.g.*, oxygen may be absorbed from underground or rain-water by contact, and thus form petroleum acids, and it may be that other chemical substances are communicated to the oil in a similar manner, the probability of which discounts

[1] This is due to the removal by the acid of the substances from which the petroleum acids are formed. More exact experiments have shown that well-refined oil on exposure to the air becomes at first lighter in color, up to a certain limit, after which point it begins to darken again, the extent of the initial transformation being in one sample of Russian oil examined, 18 mm. in ¾ hr. (measured by STAMMER'S colorimeter). Further tests were made by OSTREJKO with Baku oils examined at intervals, and stored in the dark, when the color (initially 12 mm.) registered 152 mm. The next day, the register was 115 mm., and receded to 106 mm. in another 24 hr., although protected from all light. Air was found to be absorbed throughout the whole period. Sun-bleaching is therefore an unsuitable process for Baku oils. The influence of various shades of glass on the alteration of the color of the oil was also studied. Starting with an initial color degree of 115 mm. Stammer, it was found that the maximum decoloration was reached between 3 and 9 hr., after which darkening set in.

[2] *Trudi bak. old. imp. russk. techn. obschtsch.*, **10**, (6), 1.

the value of hypotheses on the origin of petroleum based on its chemical composition.

Zaloziecki[1] has reported that in the decomposition of acid sludge, dilution, *air*, and light are the three chief factors, the air causing oxidation.

With regard to the effect of heat on the oxidation of mineral oils, it may be mentioned that Schreiber[2] has studied the effect of heating various lubricating oils to temperatures of 200°, 250°, and 280°C. in an apparatus specially constructed to obtain conditions similar to those of actual German practice in lubrication. After 16–24 hr. exposure to the high temperature, the mobility and color of the residue were noted, and determinations were made of the extent of evaporation and of the formation of bodies insoluble in petroleum spirit. It was found that high viscosity and flash-point do not necessarily mean low evaporation and the absence of resinification at high temperatures, so that it is advisable to make separate tests of all these properties. The products insoluble in petroleum spirit generally described as "asphaltic" are almost completely insoluble in hot benzene and are therefore more probably charred products. The formation of these substances apparently only takes place in an oxidizing atmosphere, so that determination, after heating, of the amount insoluble in petroleum spirit, is most important in the case of compressor oils, and not so necessary in the case of cylinder oils if the steam used is previously freed from air. The general behavior on heating was found by Schreiber to agree satisfactorily in these respects with the lubricating value in practice.

In an investigation conducted along similar lines to those followed by Schreiber, but independently of the latter, mineral lubricating oils were subjected by Waters[3] to a temperature of 250°C. for 3 hr. in open or loosely covered flasks. Each test was made upon 10 grams of oil, and, after the heating, the amount of "asphaltic matter" (insoluble in petroleum ether) and the percentage loss by evaporation were determined. It was found that, by heating duplicate samples of the same oil side by side in vessels of similar shape, size and material, fairly concordant results were generally obtained. Considerable discrepancies were, however, often caused by unequal heating, by variations

[1] *Chem. Rev. Fett- Harz-Ind.*, **5** (1898), 27.
[2] *Z. angew. Chem.*, **23** (1910), 99.
[3] *J. Ind. Eng. Chem.*, **3** (1911), 233.

in the rate of interdiffusion of oil vapor and air, and, apparently, by obscure catalytic phenomena influenced by the nature of the material in which the oil was heated. From the results obtained, Waters was of the opinion that the data afforded by the process were sufficiently reliable for the comparison of a limited number of oils with one another, as showing the way in which they might be expected to behave when in actual use.

As mentioned, the increase which takes place in the asphaltic constituents of mineral oils has been shown by Schreiber to be caused by oxidation. Meyerheim[1] has found that radium rays have no perceptible influence in increasing the proportion of asphaltum, and his experiments have shown that exposure of an oil to diffused daylight causes an increase in the amount of asphaltum insoluble in petroleum spirit. Analogous results were obtained by exposing oils to the ultraviolet rays of a mercury vapor lamp. On the other hand, with the exception of one oil, in diffused daylight the amount of asphaltic constituents insoluble in alcohol-ether was slightly reduced by exposure to either form of light.

It has been shown that the action of chlorine, sulphur, or sulphur chloride on crude petroleum leads to the formation of asphaltum. The effect of other reagents in causing polymerization and condensation has been studied by Zaloziecki and Zielinski.[2] The petroleum used was of Galician origin, with a specific gravity of 0.8539 at 15°C., a paraffin content of 1.54 per cent., carbon, 85.29, hydrogen, 14.41, sulphur, 0.19, and oxygen, 0.11 per cent. The oil was free from asphaltum. It

| Reagent | Asphaltum formed, per cent. | | | |
|---|---|---|---|---|
| | (*a*) | (*b*) | (*c*) | (*d*) |
| Anhydrous ferric chloride | 0.87 | 2.12 | 1.13 | 0.64 |
| Sodium ethoxide | 0.28 | 0.322 | 0.70 | |
| Anhydrous aluminum chloride | 1.28 | 3.46 | 7.94 | 6.28 |
| Anhydrous zinc chloride | 0.63 | 3.06 | 2.10 | 2.48 |
| Phosphorus pentoxide | 4.44 | 3.17 | 5.37 | 3.80 |
| Anhydrous cuprous chloride | 0.86 | 1.12 | 1.24 | 0.96 |
| Sulphur and aluminum chloride | ..... | ..... | ..... | 6.37 |
| Phosphorus and aluminum chloride | ..... | ..... | ..... | 6.33 |

[1] *Chem. Rev. Fett- Harz-Ind.*, **19** (1912), 28.

[2] *Orig. Com. Eighth Intern. Cong. Appl. Chem.*, **10**, 335.

was mixed with 5 per cent. of a powdered reagent, and (*a*) left for 4 weeks at the ordinary temperature in darkness; (*b*) left for 80 days at the ordinary temperature in sunlight; (*c*) heated for 166 hr. on a water-bath; and (*d*) heated to 140°C. for 35 hr. in a sealed tube. The results were as shown in preceding table.

Metallic sodium, sodium carbonate, manganese dioxide, sodium sulphide, sulphur, barium sulphide, barium peroxide, and phosphorus, were tried in some of the experiments, but with negative results. The greatest yield, 10.28 per cent., was obtained by heating to 150°–155°C. with 5 per cent. of aluminum chloride for 60 hr. The asphaltum contained 6.04 per cent. soluble in benzene, 0.14 per cent. soluble in carbon disulphide, and 4.10 per cent. soluble in chloroform. Analyses of the asphaltum by combustion, at successive dates, showed that it gradually became oxidized. The asphaltum contained a much greater proportion of sulphur than the crude petroleum. It is probable, according to Zaloziecki and Zielinski, that natural asphaltum is formed from petroleum by the action of certain mineral substances and by subsequent oxidation of the products. Sulphur compounds in the petroleum appear to take part in the condensation.

**Technical Processes.**—Since 1880, there have been patented a number of processes for the production of asphaltoid substances from petroleum. For example, De Smedt[1] claimed the treatment of coal tars and heavy petroleum oils with an oxidizing agent;[2] Hyde[3] patented an insulating compound composed of petroleum or mineral oils combined under heat, in a closed vessel, with the hard bituminous residuum from the distillation of petroleum; Waring[4] claimed an insulating material produced *by subjecting natural asphaltum, or the heavier distillates or residual products of petroleum, to a degree of heat* above the vaporizing point of water, to eliminate the latter and the light and easily decomposed products—approximately 175°C.—but below the point at which destructive distillation or cracking begins; Dubbs[5] devised a process which consisted in the com-

[1] United States Patent 239466, Mar. 29, **1881.**

[2] According to this patent, telegraph wires and electrical conductors are insulated or covered with an *oxidized hydrocarbon obtained by treating coal tars and the heavy oils of petroleum with an oxidizing agent.*

[3] United States Patent 281999, July 24, **1883.**

[4] United States Patent 284098, Aug. 28, **1883.**

[5] English Patent 3026, Feb. 16, **1892.** On sulphur processes, see Köhler's "Asphalte", **1904,** 119.

bination of crude petroleum or petroleum residues, when subjected to heat, with sulphur; Haddan[1] claimed an invention which related to the production of artificial asphaltum, or pitch, from hydrocarbon oils or their products by the addition of resinous substances, and subjecting the whole to *heat* and partial distillation; and Grey[2] devised a process wherein the retort containing a mixture of residue with limestone or the like *is heated to the requisite temperature, and while the mixture is in a heated condition, air, preferably under pressure, is admitted and efficiently diffused through the material in order to rapidly oxidize the same and bring it to the required degree of consistency.* Then, too, Forward and Davidson[3] proposed to mix crude petroleum, petroleum tar, or heavy hydrocarbon oil with sulphuric acid, then to separate the carbonaceous matter from the oils, wash the former to free it from the acid, mix with it heavy hydrocarbon oil, and subject the mixture to a high temperature[4] (preferably to 800°F.); later Forward patented a process for the manufacture of asphalt from petroleum residuum or from crude petroleum,[5] wherein petroleum residuum, having a gravity of 17°–22°Bé., is heated in a still to about 625°F. for 24 to 60 hr. in order to drive off the volatilizable constituents, and is then heated in an open vessel at a lower temperature, for example 525°F., for 5 to 24 hr., until the product has the desired consistence; and Burton[6] has claimed a process in which the liquid portions of paraffin distillates having a boiling point of upward of 500°F. (260°C.) are distilled at a temperature of from 650° to 850°F. (343°–454°C.), under a pressure of from 4 to 5 atmospheres maintained by means of the volatile constituents, until the volume of the liquid has been reduced to about 40 or 50 per cent. of its original volume, after which the liquid residue is drawn off and subjected to a temperature of from 500° to 700°F. (260°–371°C.) under substantially atmospheric pressure in an inert atmosphere until the major portion has been distilled off.

[1] English Patent 9550, May 12, **1893.**

[2] English Patent 18302, Aug. 25, **1898.**

[3] English Patent 20879, Oct. 4, **1898.**

[4] During the heating, which should be kept up from 4 to 8 hr., the conversion of the combined material into asphalt is effected.

[5] English Patent 9646, Apr. 20, **1911**; see also United States Patent 998569, July 18, **1911.**

[6] United States Patent 1055707, Mar. 11, **1913.**

Thick resins were prepared from lubricating oil residuum in Baku for the English market from 1891–1896, by driving off the more volatile portion of the residuum at about 400°, either by direct fire or superheated steam. The yield being low and the product little adapted to replace asphalt, Adiyasievitch[1] elaborated a process which was carried out at between 150° and 200° in an ordinary still by means of a blast of air with the simultaneous discharge of electricity. Treated in this manner, a residuum of specific gravity 0.930 acquired in 3 hr. a density of 1.000, with an increased flash-point. The opinion was expressed by Adiyasievitch that the thickening of the oil must be due to *chemical change.*

Kouindjy[2] discussed the process of Adiyasievitch, wherein hot air was blown into residues heated to 150°–200°C., and an electric current was caused to pass through the mass. According to him, the bitumens obtainable thereby may be used in the manufacture of asphalt.

The processes of Byerley and of Culmer are, however, the best-known commercially, and therefore merit more detailed description than the preceding.

In the manufacture of petroleum products, it had been customary, in this country, prior to 1893, to distil the crude oil in externally heated stills, so as to drive off the naphtha and the burning oil with more or less of the heavier oils, leaving a residuum or tar which could be further distilled, if desired, down to a solid body. As the distillation of petroleum residuum or tar had been commonly conducted, it had resulted, when pushed to the production in the still of a body which was solid in the still or which solidified on cooling, in the formation of a coke or a coke-containing pitch.

In accordance with the invention of F. X. Byerley,[3] petroleum residuum or tar is distilled down to a solid body by a prolonged exposure to a pitch-forming noncoking temperature, say about 600° F.,with agitation and exposure to air or analogous gas or gaseous mixture. By this means, there are produced black (or very dark brown) bodies readily soluble in petroleum naphtha,

[1] *Zap. imp. russk. tekhn. obschtsch.*, **29** (1895), 93; *J. Soc. Chem. Ind.*, **15**, 346.

[2] *Bull. de la Soc. d'encouragement pour l'industrie nationale*, **94** (1895, 1116.

[3] United States Patent 524130, Aug. 7, 1894.

say benzine of 62°Bé., in which the cokes or pitches heretofore made from petroleum were soluble at the most in comparatively small proportions. These bodies were believed to be new and were included in the patent of Byerley as new articles of manufacture, as well as their process of production. They vary, according to the extent to which the process is pushed, in hardness at atmospheric pressure (say at 60°F.) from a rubber-like consistency to a mass of a hardness and conchoidal fracture like the natural asphaltums, as, for example, Trinidad asphaltum and gilsonite from Utah. At a lower temperature the less hard bodies become harder and have a conchoidal fracture. The bodies melt at from about 200°F. to about 400°F. The higher melting bodies, say those melting at from 350°F. to 400°F., or, in other words, those which have a drying quality, are claimed to be well adapted to varnish-making, being employed in place of the natural asphaltum. These bodies may be used also for paving and roofing and for analogous purposes to which natural asphaltums are applied; but in order to melt at the temperatures which workers in those industries have found convenient to use, it is necessary, as with Trinidad asphaltum, to employ oil or the like to render them sufficiently limpid at such temperatures, and it is better therefore for such uses to employ bodies of less hardness, which have sufficient oily matter present to melt at a convenient temperature. To his product Byerley gave the name "Byerlyte", by which designation it has become widely known. It has been held[1] that there is not a prior patent or prior use relating to the production of asphaltic products from petroleum simply by the prolonged exposure to high temperature and the action of air.

In the report of an investigation on the artificial production of asphalt from petroleum, C. F. Mabery and J. H. Byerley[2] state that it has long been felt that products should be obtained from the less volatile portions of petroleum with a greater value than the coke which is the final product in the ordinary process of refining. Many attempts have, therefore, been made to conduct the final distillation in such a manner that the heavier portions may be protected from decomposition and converted into a form suitable for the preparation of asphalt or similar products.

[1] Byerley *vs.* The Sun Company, Circuit Court of the United States for the Eastern District of Pennsylvania, October Session, **1908**, No. 201.

[2] *Am. Chem. J.*, **18** (1896), 141–150.

"F. X. Byerley was the first to discover the conditions necessary to prevent ultimate decomposition, and he has perfected a method which renders possible the separation of commercial products that are serviceable in the manufacture of the various grades of asphalt, such as are used in varnishes, roofing and paving. This method has been protected by letters patent, and, under the control of a large corporation, the manufacture of these materials will doubtless assume large proportions.

"As shown by our own experiments, although the introduction of air during distillation produces greater decomposition, the first series of distillates collected under ordinary conditions, gives evidence of considerable change, especially in the higher fractions. In the Byerley method these changes are prevented to a large extent by very slow distillation. . . . The air doubtless serves several purposes. It causes oxidation to a certain extent, especially if the tar is a residue from a sulphur oil. . . . . Then the tar is protected during distillation from the decomposing action of the hot still bottom by the currents of air which keep it constantly in motion."

According to the process of G. F. and G. C. K. Culmer for the manufacture of asphaltic fluxes,[1] the oil residuum from petroleum distillation which is to be used in the preparation of asphaltic paving or roofing cements, by admixture with, say, about 80 per cent. of refined Trinidad asphalt, is first submitted to a prolonged heating (380°F.) and at the same time to the oxidizing action of air, whereby a flux is obtained which, it is claimed, mixed in even larger proportions than above stated with asphalt, renders the cement at once firm and elastic in winter without being brittle, and yet not liable to become soft on exposure to the heat of the sun—a defect of the cements prepared by mixing 80 per cent. of refined Trinidad asphalt with 20 per cent. of the oil residuum from the distillation of petroleum.

The Culmer process for the treatment of petroleum residue for producing asphalt has been described as follows:[2]

About 3½ tons of petroleum residue of 18°Bé. are placed in a hooded pan and heated to 193°C., whereupon air is blown into the contents through a perforated coil for 40 hr., the volume of air admitted per hour being 5,000–6,500 cu. ft. during the first 32 hr. and 3,000–4,000 cu. ft. during the remainder of the time. The mass is said to become thicker and an increase in the internal heat is noticeable; the loss in weight amounts to 3½–4 per cent.

[1] See United States Patents 635429 and 635430, Oct. 24, **1899**; and English Patent 11687, June 5, **1899.**

[2] *Rev. Prod. Chim.*, **2**, 356.

The changes occurring during the operation are shown in the following table giving the percentage composition of the mass at the different times specified; the increased proportion of asphaltene is said to be the cause of the suitability of the product for the purpose in view.

| | Hours | | | |
|---|---|---|---|---|
| | 0 | 16 | 32 | 40 |
| Petrolene | ..... | 90.51 | 74.40 | 73.34 |
| Asphaltene | 2.50 | 8.03 | 23.46 | 25.14 |
| Other organic matter | ..... | 0.85 | 1.40 | 0.89 |
| Mineral matter | ..... | 0.61 | 0.70 | 0.63 |

## PETROLATUM

Petrolatum[1]—defined in the "United States Pharmacopœia" as "a mixture of hydrocarbons, chiefly of the methane series, obtained by distilling off the lighter and more volatile portions from petroleum, and purifying the residue"—as prepared from American petroleum, has acquired much importance, under various trade names, as a bland neutral body well fitted to replace lard, etc., as an ointment base, for use as a protective dressing, and for lubricating purposes. It contains hydrocarbons of the paraffin series, like $C_{16}H_{34}$, $C_{17}H_{36}$, $C_{18}H_{38}$, etc., up probably to $C_{32}H_{66}$, together with hydrocarbons of the olefine series, $C_{16}H_{32}$, $C_{17}H_{34}$, etc.[2] It is stated to have a specific gravity of 0.845 at 100°F.[3]

Petrolatum is prepared from the residue left in the stills after the distillation of paraffin-base petroleum by steam; from the residue or sediment ("B. S.") deposited in tanks containing crude petroleum, of which large quantities have been collected

[1] Equivalent preparations or ones of greater or less similarity are Petroleum Ointment, *Unguentum Petrolei*, *Petrolatum Molle*, *Petrolatum Spissum*, Soft Paraffin, *Adeps Petrolei*, *Unguentum Paraffinum*, Soft Petrolatum, "Cosmoline," "Vaseline," Paraffin Jelly, Petroleum Jelly, Soft Petroleum Ointment, Hard Petroleum Ointment, Hard Petrolatum, and *Petrolatum Album*.

[2] "The Dispensatory of the United States," 19th ed., 923.

[3] LUNGE'S "Technical Methods of Chemical Analysis," 1914, **3**, i, 97.

in storage tanks in the oil regions of Pennsylvania and Ohio; or from "rod wax."[1]

**The Preparation of Petrolatum.**—The product known as "vaseline"[2] was introduced by the Chesebrough Manufacturing Company in 1871. Vaseline-like products are now made to some extent from residues, but more largely from what are termed steam-reduced amber crudes, *i.e.*, suitable crude oils from which the lighter fractions (those included under the heads of benzine, naphtha, and illuminating and lubricating oils) have been removed by steam distillation. The reducing is sometimes carefully carried out under diminished pressure, by what is termed the "vacuum process," when the "reduced" oils can be brought to 337.7°C. (640°F.) fire test without acquiring the slightest pyrogenous odor. The "reduced oils" may be oxidized by air-blowing.

The "reduced oils" are filtered through well-dried granular animal charcoal or fullers' earth in steam-jacketed filters or in chambers kept at a temperature of from 43.3° to 54.4°C. (110° to 130°F.), or in some cases higher. The first runnings from the filters are colorless, and then pass from a light straw or amber color to a color which is red by transmitted light and light green by reflected light. The latter product is known as "cylinder stock" or "filtered cylinder oil" and is collected for use as a lubricating oil. The clearer portion of the filtrate may then be brought to the proper melting point, if necessary, by the addition of steamed rod wax or amorphous paraffin, which dissolves perfectly in the warm liquid oil.

The crude oil tank residues and the solid hydrocarbons that collect in petroleum wells are purified in a similar manner. The "B. S oil" or "rod wax" is placed in a still, heat is applied, and, after the lighter products have distilled over, and indications of congelation are noticed when a portion of the distillate is allowed to cool, the heat is withdrawn, and the contents of the still, after having cooled, are transferred gradually to a percolator which contains recently heated animal charcoal. In

[1] On the utilization of the solid paraffin hydrocarbons that collect in petroleum wells in the production of vaseline, see MABERY, *Proc. Am. Acad.*, **40** (1904), 349.

[2] "Vaseline" is a proprietary name and is strictly applicable only to the product of the Chesebrough Manufacturing Company, but the term is commonly used in a generic sense.

place of bone-black, Florida fullers' earth has in recent years been used for clarification.

Petrolatum is manufactured according to the following procedure at a Warren, Pa., refinery:

The crude oil is chilled by ammonia refrigeration to about 29°F., when it yields from 25 to 50 per cent. of a heavy separate, termed "tank bottoms," which is used for the production of petrolatum, and from 50 to 75 per cent. by volume of a light portion which is used for making "bright stocks" (low cold-test cylinder stock). The "tank bottoms" are then run into fire stills and are there reduced[1] to a melting point of 100° to 120°F., after which the product is filtered through Florida fullers' earth ("clay") at a room temperature of 140°F. and is finally finished by filtration through bone-black. It is also the practice to subject the product to superheated steam after filtration through Florida earth, in order to remove the earthy odor. The filters are 6 ft. by 12 ft. and are of the gravity type, and the Florida earth is regenerated after use by retorting at a red heat following a washing with benzine and steaming dry. The yield of petrolatum by this process amounts to 20 to 30 per cent. of the "tank bottoms."

**The Commercial Varieties of Petrolatum.**—The commercial varieties of petrolatum may be classed under two heads: (1) Those which, like vaseline, are obtained as a ready formed mixture of hydrocarbons of gelatinous consistence; (2) those made by directly mixing solid paraffin of low melting point with heavy lubricating oil, such as are known in Germany as "artificial vaselines." The latter varieties are less homogeneous and are liable to deposit granules of paraffin on keeping; they are therefore not so suited for the preparation of ointments as true American petrolatum.

In warm ether, American petrolatum dissolves freely to a clear solution exhibiting a strong blue fluorescence, and the liquid remains clear, or at most becomes only slightly turbid, on cooling. On the other hand, German petrolatum is said to form a thick solution with warm ether and to give a considerable deposit on cooling. Russian petrolatum is stated to dissolve completely in warm ether and to give a clear solution which becomes turbid

[1] A current of steam is used during reduction in order to "sweeten" the product.

on cooling.[1] In the "United States Pharmacopœia" (8th rev.) there are three forms of petrolatum, as follows: *Petrolatum*, *Petrolatum Album*, and *Petrolatum Liquidum*. In the "British Pharmacopœia" there are three, as follows: *Paraffinum Liquidum*, *Paraffinum Molle*, and *Unguentum Paraffini*. The petrolatum of the "United States Pharmacopœia" (8th rev.) corresponds with the *Petrolatum Spissum* of the "United States Pharmacopœia," 1890. The *Paraffinum Molle* of the "British Pharmacopœia" resembles the *Petrolatum Molle* of the "United States Pharmacopœia," 1890, but is somewhat softer in consistence.

The "Liquid Paraffin" of the "German Pharmacopœia" is obtained from heavy fractions of petroleum by successive treatments with 66°Bé. sulphuric acid and oleum. These destroy all unsaturated constituents and leave a mixture of saturated hydrocarbons resembling melted paraffin wax.[2] In 1915, when the Russian supply was cut off, the manufacture of liquid petrolatum, or "white medicinal oil," was started on a large scale in the United States, and a number of products which are equal to "Russian white oil," are now being marketed for use, principally in the treatment of constipation.[3]

**Official Petrolatum.**—The "United States Pharmacopœia" describes petrolatum as follows: "An unctuous mass, of about the consistence of an ointment, varying in color from yellowish to light amber, having not more than a slight fluorescence, even after being melted, transparent in thin layers, completely amorphous; without odor or taste, but giving off, when heated, a faint petroleum-like odor. If a portion of petrolatum be liquefied and brought to a temperature of 60°C. (140°F.), it should have a specific gravity of from 0.820 to 0.850. Petrolatum is insoluble in water; scarcely soluble in cold or hot alcohol, or in cold absolute alcohol, but soluble in boiling absolute alcohol, and

[1] ALLEN'S "Commercial Organic Analysis," 2nd. ed., **2**, 408. Russian vaseline is an artificial product, prepared from liquid vaseline oil and ceresin (STAZENKO, *Farm. Westnik*, **4** (1900), 376).

[2] See *Oil of Paraffin* (p. 891), *Perfumery Oil* (p. 896), *Petrolatum (Liquid)* (p. 898), and *Petrolatum Oil* (p. 899). For a general account of medicinal white oils, see *Nat. Petrol. News*, **7** (1915), No. 6, 46. For a chemical consideration of pharmaceutic naphthene oil (liquid petrolatum), see BROOKS, *J. Am. Med. Assn.*, **66** (1916), 24. BROOKS discusses the testing of oils of this class.

[3] On clinical experience with liquid petrolatum, see BASTEDO, *J. Am. Med. Assn.*, Mar. 6, **1915**, 808.

readily soluble in ether, chloroform, carbon disulphide, oil of turpentine, petroleum benzine, benzene, and fixed or volatile oils. The melting point of petrolatum ranges between 45° and 48°C. (113° and 118.4°F.). If heated on platinum foil to a still higher temperature, petrolatum should be completely volatilized without emitting any acrid odor. If melted petrolatum be well shaken with water, the latter should not redden blue litmus paper. If 10 grams of petrolatum be digested at 100°C. (212°F.) for ½ hr. with 10 grams of sodium hydroxide and 50 c.c. of water, the aqueous layer separated and supersaturated with sulphuric acid, no oily or solid substance should separate (absence of fixed oils or fats of animal or vegetable origin and of rosin)." The "British Pharmacopœia" describes it as "white or yellow, translucent, soft, unctuous to the touch, free from acidity, alkalinity, or any unpleasant odor or flavor, even when warmed to 120°F. (48.9°C.). Specific gravity at the melting point, 0.840 to 0.870. Melts at 96° to 102°F. (35.5° to 38.9°C.) or even somewhat higher, volatilizes without giving off acrid vapors, and burns with a bright flame, leaving no residue. Insoluble in water, slightly soluble in absolute alcohol, freely soluble in ether, chloroform and benzol. After treating with a boiling solution of sodium hydroxide, the aqueous liquid yields no precipitate or oily matter on adding excess of acid (absence of fixed oils, fats, and resin)." As it is found in the market, petrolatum varies in color, odor, and melting point. The statement frequently made by the manufacturers that it is unalterable in the air is incorrect, as it has been frequently noticed that exposure to light and air causes it to assume a disagreeable odor resembling that of crude petroleum.[1] It has been observed to have at times irritating properties, which may be due to imperfect removal of the sulphuric acid or similar agents used, but are more probably caused by rancidity or decomposition.

According to Mabery,[2] vaseline consists of heavy oils of the series $C_nH_{2n}$, $C_nH_{2n-2}$, and $C_nH_{2n-4}$, with solid paraffin hydrocarbons, the latter being sufficient in quantity to saturate the former and produce an emulsion.

**The Preparation of German Petrolatum.**—Petrolatum is obtainable not only from the paraffin-base petroleums of the

[1] The Dispensatory of the United States," 19th ed., 923.

[2] *Proc. Am. Acad.*, **40** (1904), 361.

United States, but also from the oils of Galicia,[1] Elsass[2] and Russia.[3]

According to German practice,[4] the oil under treatment is (1) heated by steam to about 30°C. (86°F.), mixed, at this temperature, with 10 per cent. of its weight of sulphuric acid of 60°Bé., stirred for ½ hr., and then allowed to stand at rest, so that the carbonized portions may separate. (2) When clear, the oil is washed with a water solution of potassium dichromate, whereby any remaining excess of sulphuric acid is removed. (3) The residue from the acid treatment is mixed with lime, neutralized, and disposed of to fertilizer factories. (4) The clear oil from the second step of the process, after being washed, is heated by steam to 80°C. (176°F.), mixed with 10 per cent. of its weight of granular animal charcoal, and then allowed to stand at rest, to permit the animal charcoal to settle. (5) After the latter is separated, the liquid portion is filtered through filters heated by steam. (6) The residuary magma of animal charcoal is subjected to hydraulic pressure, the expressed oil filtered, and the solid residue is again used in the next operation, a sufficient quantity of fresh animal charcoal being added to make up for any loss or waste.

**The Chemistry of German Petrolatum.**—Petrolatum obtained from Galician petroleum has been investigated by Engler and Böhm,[5] who studied two especially suitable crude oils from Ropa, in Galicia.[6] On account of their technical importance, the results of this inquiry are presented at some length below.

Two methods were employed by Engler and Böhm for obtain-

[1] ENGLER and BÖHM, *Dingler's polyt. J.*, **262** (1886), 468.

[2] *J. Russ. Phys.-Chem. Soc.*, **13**, 454.

[3] MENDELÉEFF, *Fortschr. Jahresber. Chem.*, **1882**, 1458; BIEL, *ibid.*, 1466; and STAZENKO, *Farm. Westnik*, **4** (1900), 376.

[4] *Pharm. Centralh.*, **1881**, No. 42; *New Remedies*, February, **1882**.

[5] *Dingler's polyt. J.*, **262** (1886), 468 and 524.

[6] These oils, which were strongly dichroic, gave the following results on fractionation:

| | Sp. gr. at 15°C. | Fraction under 150°C. | | 150° to 290°C. | | 290° to 340°C. | | Over 340°C. | |
|---|---|---|---|---|---|---|---|---|---|
| | | Volume, per cent. | Weight, per cent. | Volume, per cent. | Weight, per cent. | Volume, per cent. | Weight, per cent. | Volume, per cent. | Weight, per cent. |
| No. I..... | 0.812 | 30.2 | 26.7 | 35.9 | 35.5 | 5.3 | 6.5 | 27.7 | 31.1 |
| No. II..... | 0.820 | 21.8 | 20.0 | 51.7 | 51.2 | 8.8 | 9.4 | 17.0 | 18.9 |

ing the vaseline—the first consisting in dissolving the residue in petroleum ether, decolorizing the solution by bone-black, and removing the solvent by evaporation; and the second in bleaching the oil itself, and evaporating it to the consistency of vaseline.

1. *Preparation from Residue.*—When the residue, after expelling the portions with boiling points up to 340°C., became syrupy, about one-third more was expelled, the remainder having about the consistency of butter. One part of this was dissolved in 7 parts of petroleum ether of 0.66 specific gravity, and treated repeatedly with one and a half times its weight of powdered bone-black for 1 or 2 hr. After the seventeenth treatment the fluorescent solution became water-white. On evaporating off the solvent, the vaseline was left as an odorless, tasteless, and colorless oil, with strong blue fluorescence, congealing to a translucent mass consisting solely of hydrocarbons and melting at 32°C. It showed no crystalline structure, nor did the precipitate produced on cooling down from solution in hot alcohol display any crystalline appearance. On the other hand, paraffin scale separated in quantity out of the distillates from the concentration of the residue.

2. *Preparation from Decolorized Crude Oil.*—The oil was bleached by passage through bone-black filters, similar to those used in sugar refineries, 75 cm. high and 5 cm. wide, provided with a steam jacket, and containing 0.8 to 1 kg. of bone-black. Usually six of these filters sufficed to decolorize the oil, leaving it fluorescent and with a specific gravity reduced from 0.812 to 0.795 in the case of oil No. I, and from 0.820 to 0.782 in that of oil No. II. Distillation *in vacuo* (10 to 15 mm. mercury column) at 250°C. was effected in a flask placed up to the neck in a Lothar Meyer air-bath. The resulting colorless, odorless, and translucent vaselines had the following properties.

| | Yield, per cent. | Specific gravity at 20°C. | Melting point |
|---|---|---|---|
| Oil I..... ..... | 13.8 | 0.8809 | 30° to 31°C. |
| Oil II..... | 13.2 | 0.8785 | 30° to 31°C. |

The composition of the vaselines obtained respectively from the residues and direct from two crude oils, shown in the following table, indicates that the products are pure hydrocarbons:

TABLE LII.—COMPOSITION OF GALICIAN VASELINES

| Percentage composition | From the residues | | From No. 1 oil | | | From No. 2 oil | |
|---|---|---|---|---|---|---|---|
| | I | II | III | IV | V | VI | VII |
| C | 86.99 | 86.67 | 86.30 | 86.54 | 86.55 | 86.14 | 86.17 |
| H | 13.14 | 13.15 | 13.99 | 13.73 | 13.74 | 13.50 | 13.72 |
| Mean atomic composition | | | | | | | |
| C | 7.23 | | 7.20 | | | 7.18 | |
| H | 13.14 | | 13.82 | | | 13.61 | |

In order to learn whether the melting point of the products could be raised by distilling off a portion, and whether the chemical nature of the vaseline was affected thereby, samples of the vaseline from the bleached oils were fractionally distilled until only a few drops of residue remained. The following results show that the melting point of the distillates increases progressively:

MELTING POINTS OF VASELINE DISTILLATES

| | | |
|---|---|---|
| Vaseline from I..... | Fraction 250° to 270°C.: 22° to 23°C. | 290° to 330°C.: 27°C. |
| Vaseline from II..... | 24°C. | 290° to 310°C.: 28°C. |

The melting point of the residue rises, after parting with the first fraction, from 30.5° to 32° to 33°C., subsequently falling, and becoming, after removal of the second fraction, 29° in the case of No. I and 27° in the case of No. II. This was probably the result of decomposition, and it is noteworthy that the distillates became crystalline in structure, while the residual vaseline in the still retained its amorphous character to the last.

The distillate resembled a mixture of paraffin and volatile oils; and since, owing to the softness of the paraffin and the consistency of the oil, separation could not be effected by filtration, absorption, or pressure, Engler and Böhm adopted a method of fractional precipitation from an ethereal solution by means of alcohol. In this process the distillate was dissolved in the minimum quantity of ether in the cold, the paraffin precipitated by agitation with absolute alcohol, separated by filtration, washed with alcohol, and dried. The process was repeated on the filtrate until all the solid paraffin was eliminated, and oil drops

were precipitated, when the liquid was evaporated, and the residue treated with minimum quantities of ether and alcohol on cooled filters until oily drops again appeared. The solids were then treated again by the same method to secure perfect separation.

In order to determine the accuracy of the process, experiments were made with 40 grams of a mixture consisting of 30 parts of "paraffinum liquidum" and 10 parts of paraffin of 56°C. melting point. Satisfactory results were obtained, but whether the method would be equally effective in the case of a mixture of very soft paraffin with very thick oil, and under other experimental conditions, was not ascertained. Fractions I to IV of the vaseline No. I from the distillation, gave by this process, with four precipitations:

TABLE LIII.—PARAFFINS IN VASELINE DISTILLATES

| | Fraction, °C. | Melting point, °C. | Melting points of the precipitates | | | | Remainder |
|---|---|---|---|---|---|---|---|
| | | | 1 °C. | 2 °C. | 3 °C. | 4 (In the cold) °C. | |
| I | 250° to 270° | 22° to 23° | 50° to 51° | 41° to 42° | 35° to 36° | 35.5° to 36.5° | Fluid. |
| II | 270° to 290° | 28° to 29° | 41° to 42° | 41° to 42° | 23° to 24° | 43° to 44° | Fluid. |
| III | 290° to 330° | 27° | Too small to estimate | | | | |
| IV | (About half over 330°) | 30° to 31° | 55° to 56° | 48° to 49° | 35° to 36° | 45° to 46° | Fluid. |

NOTE.—The paraffin precipitated from the cold mixture (4) was always of higher melting point than the preceding precipitates obtained at the temperature of the laboratory.

The same vaseline, undistilled, gave the following results:

| | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Melting point of precipitates.... | 51°C. | 49°C. | 40° to 41°C. | 29° to 30°C. | In the cold 35° to 36°C. |

None of these latter precipitates presented a crystalline structure, even after remelting, while the precipitates from the fractions were all crystalline. Similar results were obtained with the No. 2 vaseline and its distillates.

For the purpose of separating the solids from the liquids without consideration of the melting points, the same process was pursued, but with a larger quantity of alcohol, until only the fluid oil remained. As the result, 100 grams of vaseline No. 2, melting at 30° to 31°, yielded 40.8 grams of solids, of specific gravity 0.8836 (at 20°), melting at 40°C. This was termed "solid vaseline," to distinguish it from true paraffin. The

remainder, the "liquid vaseline," was of specific gravity 0.8809, and only began to solidify at −10°. That the whole vaseline should possess a specific gravity lower than that of either of its constituent parts is not unprecedented, Grotowsky[1] having observed the same thing in mixtures of paraffin and oil. The following shows the composition of the solid and the liquid vaseline:

| | Solid vaseline | | Liquid vaseline |
|---|---|---|---|
| C | 86.17 | 86.34 | 86.47 |
| H | 13.85 | 13.73 | 13.60 |

Both the solid and liquid vaseline boil at 240° to 245°C., and at 340° yield a distillate of twice the amount of the residue left in the case of the solid, and three times the amount in the case of the liquid vaseline. The residue of the solid vaseline is amorphous, and melts at 40° to 41°, while the distillate, which is crystalline, melts at 36° to 37°C.

The residue and the distillate from the fluid vaseline are liquids, the former being as thick as heavy Russian oleonaphtha. To ascertain which part of the vaseline changed from the amorphous to the crystalline form, and whether a change in the melting point is accompanied by a variation in composition, Engler and Böhm distilled solid vaseline of 40° melting point, *in vacuo* to a few drops of residue, obtaining a distillate congealing to a wax-yellow solid which melted at 37°C. After being bleached with bone-black, the composition was found to be 85.98 and 86 per cent. carbon to 14.10 and 14.16 per cent. hydrogen. On congealing after fusion, this vaseline contracted considerably, a property observed in paraffin, but not usually possessed by vaseline. Compared with solid vaseline—average 86.25 per cent. of carbon and 14.17 per cent. of hydrogen—this product shows a not inappreciable increase of hydrogen, and a slight diminution of carbon, which may be explained by the supposition that distillation separates portions richer in hydrogen from those richer in carbon (the residue), or that, by a process of dissociation, two sets of compounds are formed, one richer, the other poorer in hydrogen. Engler and Böhm incline to the former opinion, and attribute the crystallization of the hydrocarbons to a transformation from the amorphous to the crystalline state. American vaseline of 32° to 33° melting point similarly fractionated, yielded only 14 per cent. of "solid vase-

[1] *Z. Berg.-, Hütt.- u. Salinenwesen*, **24** (1876), 42.

line," of 49° to 50°C. melting point, the remainder being fluid at ordinary temperatures.

Natural vaseline is thus shown to consist of a mixture of solid and fluid hydrocarbons, both, so far as concerns the product from Galician oils, of almost identical percentage composition and boiling point. These hydrocarbons are, on the whole, saturated compounds. A drop of bromine added to the fluid oils, or to the solution of the solid vaseline in carbon bisulphide, produces a rapid evolution of hydrobromic acid, which would not be the case with homologues of the ethylene and acetylene group. The presence or absence of aromatic hydrocarbons was not determined. The solid hydrocarbons are amorphous before distillation, and it would seem that no crystalline paraffins exist, as a rule, in crude petroleum, but that the solid members of the series are more of the nature of ozokerite.

It is apparent, from the preceding researches, that considerable differences exist between natural and artificial vaseline, the latter becoming more readily granular, and separating comparatively easily into oil and paraffin. The viscosity also differs, as is shown by the following results obtained with Engler's apparatus,[1] water being taken as the unit:

VISCOSITY OF VASELINE

| | At 45°C. | At 50°C. | At 80°C. | At 100°C. |
|---|---|---|---|---|
| Natural American vaseline.... | 4.8 | 3.7 | 2.1 | 1.6 |
| Artificial American vaseline.... | Did not flow. | | 1.5 | 1.2 |

Differences are also observed in their behavior on heating and recooling: natural vaseline changing gradually from its pasty consistency into a fluid of regularly decreasing viscosity, these changes being reversed on cooling; while artificial vaseline, after becoming somewhat softer, suddenly assumes the condition of a thin fluid, and, on recooling, parts of the solids separate out.

Regarding the acids present in vaseline, those produced during the refining of the oil, such as sulphonic acid, must be distinguished from those formed by the action of the air. Engler and Böhm consider that the best method of preparing artificial vaseline quite free from acid, is that of the "German Pharmacopœia;" while for natural vaseline, the method previously described, of simple decolorization of petroleum residue by bone-black, is decidedly preferable to any other.

[1] *Dingler's polyt. J.*, **258** (1885), 126.

Fresenius[1] experimented on the acidifying action of oxygen on vaseline by heating in a glass tube 4.17 grams of natural vaseline (Chesebrough Manufacturing Company) for 15 hr. at 110°C. with oxygen, of which 21.8 c.c. were absorbed, while 4.08 grams of German "Virginia vaseline" absorbed only 3.2 c.c. of oxygen. In the former case, the subsequent ethereal solution of the oxidized vaseline gave a decided reaction, while in the latter the indication of the presence of acid was very slight.

Since the report of this experiment did not indicate the amount of acid produced, and did not state whether the Virginia vaseline was prepared according to the "German Pharmacopœia," Engler and Böhm experimented with 11 to 15 grams of vaseline, which were mixed with 2 to 3 c.c. of water in order that the oxidation might be facilitated by the presence of steam, the mixture being heated with the proportions of oxygen given in the appended table, in sealed glass tubes for 24 hr. at 110° to 115°C. The quantity of oxygen absorbed was measured by breaking the end of the tube under water, and the acid in the water which had been mixed with the vaseline, and in an ethereal solution of the vaseline, was determined by alcoholic $\frac{1}{50}$-normal potassium

TABLE LIV.—OXIDATION OF VASELINE

| Substance | Quantity employed | | Results | | |
|---|---|---|---|---|---|
| | Vaseline, etc., grams | Oxygen, c.c. | Oxygen absorbed, c.c. | Amount of acid resulting. Expressed in KOH | |
| | | | | Mg. | Weight, per cent. |
| Natural vaseline, I | 12.9 | 59.5 | 34.9 | 5.5 | 0.043 |
| Natural vaseline, II | 11.6 | 71.5 | 46.5 | 9.1 | 0.078 |
| Natural vaseline, III | 14.7 | 76.3 | 42.9 | 10.5 | 0.071 |
| Artificial vaseline, I | 15.5 | 74.0 | 4.5 | 0.7 | 0.005 |
| Artificial vaseline, II | 11.7 | 69.1 | 4.7 | 0.7 | 0.006 |
| Artificial vaseline, III | 14.8 | 74.7 | 4.2 | 1.4 | 0.009 |
| Lard, I | 14.5 | 71.2 | 49.8 | 31.1 | 0.214 |
| Lard, II[2] | 14.9 | 57.3 | 42.0 | 39.4 | 0.264 |
| Vaseline oil, I | 12.0 | 56.7 | 5.5 | ..... | ..... |
| Vaseline oil, II | 11.0 | 56.9 | 4.1 | ..... | ..... |
| Ceresin | 11.4 | 52.7 | 3.0 | ..... | ..... |

[1] *Idem*, **236** (1880), 503.

[2] The acidity of the lards before the experiment was taken into consideration in this result. The vaselines were free from lard.

hydroxide. The same experiment was carried out with artificial vaselines (1 part of ceresin mixed with 3 parts of two kinds of vaseline oil), prepared according to the formula of the "German Pharmacopœia," as well as with the component parts of these mixtures, and with two samples of lard.

Two 10-gram samples of the natural vaseline above referred to, spread out on a glass plate over an area of 16 sq. cm., and exposed to the air for 14 days, near the fire (at a temperature of about 30°C.), yielded the following results, in comparison with artificial vaseline and lard similarly treated:

| | Natural vaseline | Natural vaseline | Artificial vaseline | Lard |
|---|---|---|---|---|
| | I | II | | |
| Per cent. weight of acid, expressed in mg. of KOH.. | 0.025 | 0.026 | 0.015 | 0.48 |

Warmed from 40° to 50°C. for 2 days in a closed glass tube containing air, the results were:

| | Natural vaseline | Artificial vaseline | Lard |
|---|---|---|---|
| Oxygen absorbed... | 2.0 c.c. | 1.5 c.c. | 2.3 c.c. |

No difference was observed when oxygen was substituted for air.

For all purposes where lubricating properties and viscosity at somewhat elevated temperatures are required, the natural vaseline is said to be preferable to the artificial;[1] but the reverse appears to be the case where fluidity at a moderate temperature is desired and where an appreciable tendency to acidification is prejudicial.

[1] For a rather full account of the "artificial vaselines" which are prepared by dissolving paraffin in hot vaseline oil or heavy oil of the density 0.850–0.860 which has been deodorized and decolorized, see GIRARD, *J. Pétr.*, **3** (1903), 619. On distinguishing artificial and natural vaselines, see ARMANI and MELLANA, *Giorn. farm. chim.*, **62**, 121.

## CHAPTER XIV

## REFINERY ENGINEERING

In treatises of the usual type devoted to petroleum technology, complete working drawings, such as will be found in this chapter, are, to say the least, quite infrequent. In fact, the authors of handbooks on the petroleum industry have rarely, if ever, provided the refiner or refinery engineer or the student of petroleum engineering with such drawings and specifications as will be of assistance to him in the designing of a plant for the treatment of crude oil or the remodeling of an old refinery.

An attempt has been made by the authors of the present work to meet the existing demand for information regarding the design, erection and equipment of petroleum refineries. The drawings included in the following pages embody the result of the experience of American experts in refinery engineering. The authors do not, of course, make a claim to finality in presenting the various arrangements suggested, but the refiner and student who familiarize themselves with the drawings and specifications are assured that the practice which is therein indicated has been followed with success in existing American petroleum refineries. In the nature of things it is not possible to furnish accounts of what are still semi-secret mechanical appliances. It is likewise out of the question to supply detailed descriptions of types of refinery plant which are still in course of development. But it is the hope of the authors that the following presentment of sound practice in up-to-date construction will be found of value by those responsible for the management of petroleum refineries and of interest to the chemical engineering profession.

### GENERAL SPECIFICATIONS

It is the experience of eastern refiners that a refinery capable of handling 10,000 bbl. of crude oil per month, or 400 bbl. per day, including the necessary pressing, filtering and barreling houses, but exclusive of tank cars, costs $100,000. For purposes of estimation, it may therefore be stated that, according to Penn-

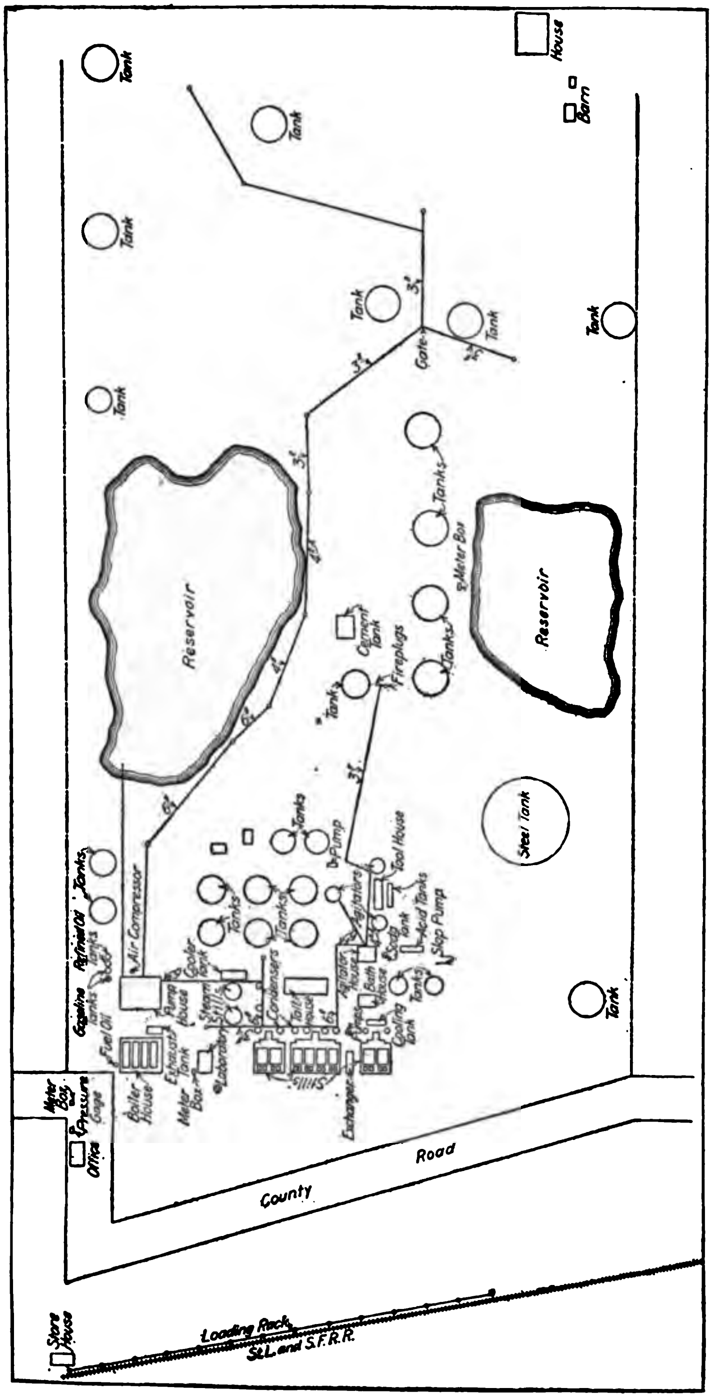



**Fig. 207.—Layout of the plant of the Sapulpa Refining Company, Sapulpa, Okla. (Courtesy of W. D. Richardson, Sapulpa, Okla.)**

sylvania practice, the cost of refining plant is about $20,000 per million gallons of crude oil put through annually.

The following specifications for refinery iron work have been contributed by W. R. Schellhammer, of the Hammond Iron Works, Warren, Pa. These cover the stills, condensers, agitators, tankage, bleachers, blow cases, and filters required for (1) a refinery at Eldred, Pa., capable of handling 15,000 bbl. of petroleum per month (see Fig. 209); and (2) a refinery at Chanute, Kan., having a throughput of 175,000 bbl. of petroleum per month. The first-mentioned was erected in 1913, the latter in 1914.

### 1. Specifications for a Refinery at Eldred, Pa.

#### Three Crude Stills, 10 ft. by 34 ft.

**Size and Style.**—These stills will be of the horizontal type, ten feet in diameter by thirty-four feet long (10 ft. by 34 ft.) in the shell.

**General Construction.**—The bottom of these stills will be made in one solid plate, one hundred and eight inches (108 in.) wide, running the full length of the still.

The upper part of the shell will be made in five courses running girthwise, one sheet to each course or ring. Each of the mainheads will be made in one solid plate and will be dished outward and flanged-in.

**Dome.**—On the center course of the stills there will be a dome, 36 in. in diameter by 36 in. high; the shell of the dome and the head of the dome are each to be made in one plate, the dome head to be dished upward and flanged-in.

**Steel Plate.**—These stills will be built throughout of the best quality open-hearth homogeneous flange steel, with the exception of the long bottom plate, which is to be made of fire-box steel.

All the steel entering into the construction of these stills shall conform to the standard specifications adopted by the Association of American Steel Manufacturers.

Bottom of stills to be of ½-in. steel.
Shell of stills to be of ⅜-in. steel.
Heads of stills to be of 7/16-in. steel.
Dome of stills to be of ⅜-in. steel.
Dome head of stills to be of ⅜-in. steel.

**Riveting.**—These stills will be punched for double riveting throughout with ¾-in. diameter rivets, with the exception of the dome head, manhole neck, flanges, etc., which will be punched for single riveting.

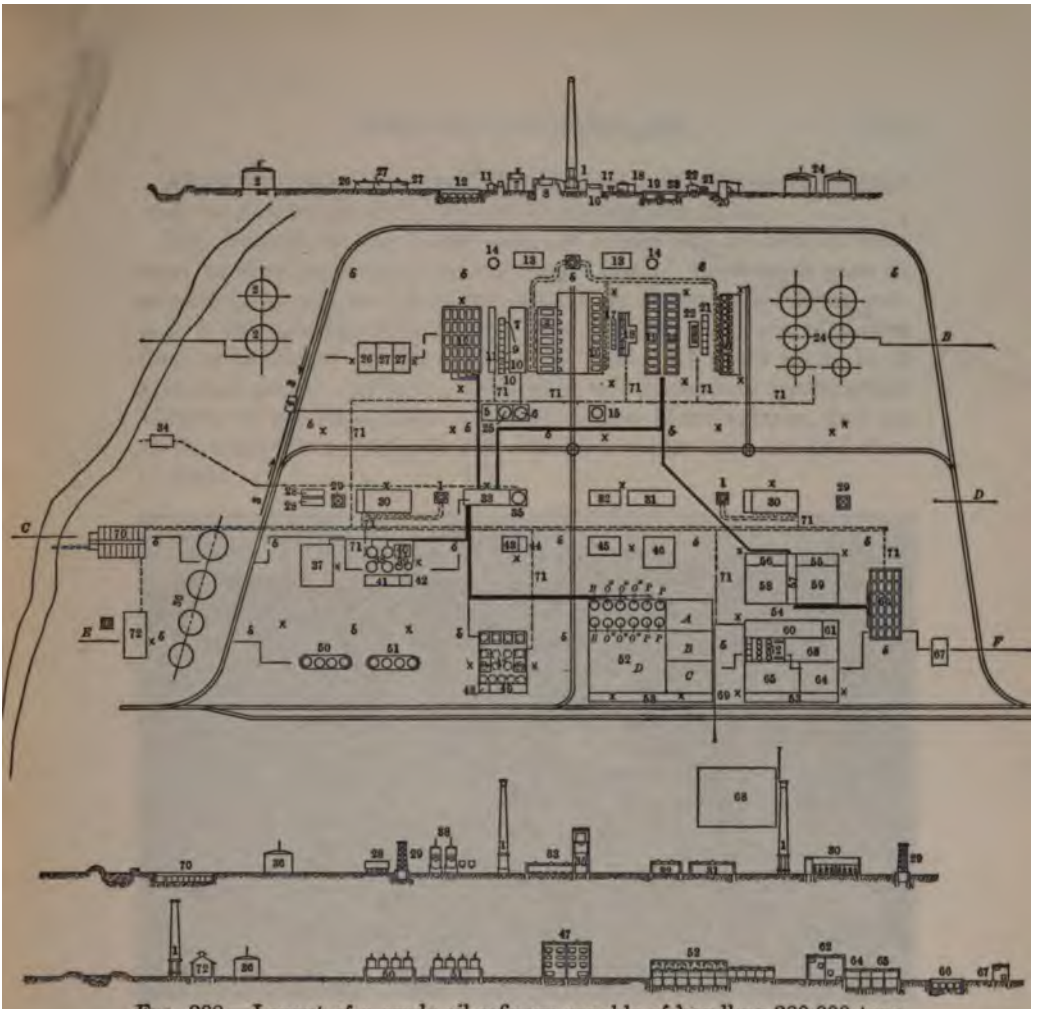


FIG. 208.—Layout of a crude oil refinery capable of handling 200,000 tons of paraffin-base petroleum. (After Engler and Höfer.)

1. Chimneys.
2. Crude oil supply tanks.
3. Crude oil pipe-line.
4. Crude oil pump-room.
5. Crude oil pump-house.
6. Central preheater.
7. Preheating battery.
8. Petrol stills.
9. Petrol coolers.
10. Benzine coolers.
11. Valve room.
12. Petrol receivers.
14. Emergency receivers.
15. Central preheater by oil still battery.
16. Oil stills.
17. Oil condensers.
18. Oil distillation.
19. Oil receivers.
20. Cracking stills.
21. Cracking coolers.
22. Cracking distillation.
23. Cracking receivers.
24. Tanks for intermediates.
25. Filling tower.
26. Boiler and tank shop.
27. Repair shops.
28. Acid and alkali storage.
29. Cooler.
30. Water flushing plant.
31. Engine house.
32. Electrical central station.
33. Electrical pump-house.
34. Water station.
35. Water tower.
36. Supply tanks.
37. Benzine fractionation.
38. Petrol agitators.
39. Benzine agitators.
40. Acid and alkali waste montejus.
41. Petrol filter.
42. Benzine pump-house.
43. Fire department.
44. Telephone central.
45. Magazine.
46. Laboratory.
47. Lubricating oil refinery.
48. Pumping house.
49. Filling room.
50. Benzine reservoir.
51. Petrol reservoir.
52. Cooperage.
A. Cooperage and gluing room.
B. Drying room.
C. Painting room.
D. Filling and shipping room
P. Petrol reservoirs.
O. Oil reservoirs.
*B*. Benzine reservoirs.
53. Shipping platform.
54. Paraffin plant.
55. Gas motor.
56. Reserve engine.
57. Pump-house.
58. Storage.
59. Storage.
60. Sweating room.
61. Pump-house.
62. Refinery.
63. Filling room.
64. Packing room.
65. Shipping room.
66. Filtrate receivers.
67. Extractors.
68. Barreling room.
69. Barrel elevator.
70. Waste water purifier.
71. Waste water lines.
72. Waste acid regenerating plant.

X = Hydrants.

δ = Arc lamps.

(*Facing page* 646.)



All rivets must completely fill the holes and have full heads concentric with the body of the rivet, of a height not less than ½ in.

The diameter of the punch shall not exceed 1/16 in. and all holes must be clean cut without torn or ragged edges. Rivet holes must be accurately spaced, the use of drift pins being allowed only for the purpose of bringing members together, and must not be driven with such force as to disturb the metal about the holes. All rivets are to be of first-class quality and make, conforming to the standard specifications adopted by the Association of American Steel Manufacturers, and are to be spaced in accordance with the best known rules for this class of work.



FIG. 209.—Petroleum refinery at Eldred, Pa.

**Caulking and Testing.**—All plates are to be neatly and carefully bevel-sheared on a rotary beveler for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps.

**Openings.**—On the top of the shell of these stills at the back end, or other approved location, there will be one 20-in. diameter cast-iron still manhole, provided with cover complete, crossbar and screw. On the front head of the stills, will be a 20-in. diameter steel manhole neck, finishing at the outer end with a cast-iron mangle ring and bolted cast-iron cover secured with ¾-in. diameter square-shouldered bolts; a wrought-iron crane for carrying the cover is also to be furnished and attached to the manhole neck. The neck is to be made of flange steel plate.

These stills will be provided with the following forged steel pipe flanges, all to be taper-threaded for standard pipe connections of the sizes given:

One 10-in. flange located on the top of dome.
Two 4-in. flanges located on the top of shell.
One 4-in. flange located on the bottom of shell, at the back end.

If preferred, there will be furnished, instead of the 4-in. flange in the back end of the bottom, one 4-in. diameter cast-iron outlet nozzle having a wrought-iron rod extending up through the shell of the still and passing through a cast-iron stuffing box riveted on the shell of still in line with outlet nozzle beneath; to the bottom of this tar plug outlet nozzle there will be bolted one cast-iron flange, taper-threaded for 4-in. pipe. The tar plug and seat will be ground joint.

**Castings.**—For supporting the stills on masonry, there will be furnished twelve heavy cast-iron brackets. These brackets are to be cored for 1-in. diameter rivets.

**General.**—These stills are to be built complete in the shop, tested when full of water, and made absolutely tight and dry. The dome and brackets are to be shipped seperately and riveted in place at the destination.

## One Steam Still

**Size and Style.**—This steam still will be of the horizontal type, ten feet in diameter by thirty-four feet long (10 ft. by 34 ft.), center to center of the rivets in the end seams.

**General Construction.**—The shell of this still will be made in five roundabout courses, each 84 in. wide. Each end of the still will be made of one plate and will be dished outward and flanged-in.

**Steel Plate.**—The shell of this still will be built throughout of open-hearth homogeneous tank steel, the heads of the flange steel conforming to the standard specifications adopted by the Association of American Steel Manufacturers. The shell of the still is to be made of $\frac{3}{8}$-in. steel; the heads of $\frac{3}{8}$-in. steel plate.

**Riveting.**—This still will be riveted throughout with $\frac{3}{4}$-in. diameter rivets, spaced in accordance with standard practice. Girth seams will be single; horizontal seams, double-riveted.

**Caulking and Testing.**—All plates are to be neatly and carefully sheared for caulking on the outside, and the corners are to be scarfed to insure tight joints at the laps.

The still is to be thoroughly caulked on the outside, tested when full of water, and made absolutely tight and dry.

**Openings.**—On the top of the shell of this still, there will be one 20-in. diameter cast-iron manhole, complete with cover, crossbar and screw. In the front head of the still, there will be one 20-in. diameter manhole neck with steel angle ring and steel plate bolted cover. Riveted to the shell of the still at an approved location there will be five 6-in. forged steel flanges. There will also be two 3-in. forged steel flanges, placed where desired by the purchaser.

**General.**—This still is not to be provided with brackets. It is to be built complete in the shop, tested when full of water, given one coat of paint on the outside, and shipped intact.

## Four Condensers, 40 ft. by 10 ft. by 8 ft.

**Size and Style.**—These condensers will be rectangular in shape, forty feet long, ten feet wide and eight feet deep (40 ft. by 10 ft. by 8 ft.), open top.

**General Construction.**—Each condenser will be constructed in eight courses, each 5 ft. wide. Each course is to be made in two plates, bent at each lower edge to a radius of 4 in., so as to form the sides of the condenser.

Each end of the condenser will be made in two plates and flanged-in.

**Angles and Bracing.**—For the purpose of stiffening each condenser, there will be furnished, for around the top on the outside, an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle. This angle will not be welded, but will have split joints neatly butted together with covering shoes.

For riveting to each course of the condenser, there will be two vertical stiffeners of 3-in. by 3-in. by ⅜-in. steel angles, eight to each side set opposite each other. For riveting to each end of the condenser, there will be furnished two vertical angles of the same size.

For further bracing of this condenser, there will be provided eight braces of 2-in. by ¼-in. steel bar, running crosswise of the condenser midway between the top and bottom, with each end punched for bolting to the vertical angle irons.

There will also be furnished two braces in each head, or end, punched for bolting at one end to the vertical angle iron that is furnished for the head, the other end being punched for riveting to the nearest end of the condenser. Running crosswise of the condenser, straight and diagonally, there will be furnished a set of braces of 2½-in. by ¼-in. flat steel bar, punched for bolting at each end to the top angle iron.

**Steel Plate.**—The steel plate used in the shell, that is, the sides and bottom of each condenser, will be the best quality No. 6 B.W.G. tank steel, and that used in the heads or ends of the best quality open-hearth homogeneous flange steel, No. 6 B.W.G. All the steel used in the construction will conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—Each condenser will be riveted throughout with ⅜-in. diameter rivets, spaced 1¼ in. center to center, single-riveted.

**Caulking and Testing.**—All the plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps.

**Openings.**—There will be furnished one 4-in. and one 2-in. pipe flanges, which flanges are to be of forged steel, straight threaded.

**Painting.**—On completion, the condensers are to be tested, made absolutely tight and dry, and painted with one coat of good mineral paint on the outside.

### One Cooling Box, 20 ft. by 6 ft. by 4 ft.
### One Cooling Box, 20 ft. by 6 ft. by 6 ft.

**Size and Style.**—These boxes will be of the rectangular shape, one twenty feet long, six feet wide and six feet deep (20 ft. by 6 ft. by 6 ft.), and one twenty feet long, six feet wide and four feet deep (20 ft. by 6 ft. by 4 ft.). They are to be open top.

**General Construction.**—These boxes will be constructed in four courses, each 5 ft. wide. Each course is to be made in two plates, bent at each lower edge to a radius of 6 in., so as to form the sides. Each end of the boxes will be made in one solid plate, flanged-in.

**Angles and Bracing.**—For the purpose of stiffening these boxes, there will be riveted around the top of the outside an angle 2½ in. by 2½ in. by 5/16 in. It will not be welded but will have split joints neatly fitted together with covering shoes. Riveted to each course of the boxes there will be vertical stiffeners of 3-in. by 3-in. by ⅜-in. angle, set opposite each other. Riveted to each end of the boxes there will be two vertical angles of the same size.

The boxes will be further braced by a 2-in. by ¼-in. steel bar running crosswise, each end bolted to the vertical angle irons. There will also be two braces in each head, bolted at one end to the vertical angle iron that is riveted to the head and the other end to the nearest end of the boxes.

**Steel Plate.**—The steel plate used in these boxes will be ¼ in., weighing 10.2 lb. to the square foot throughout. The shell proper is to be of tank quality and the head of flange-quality steel plate. All plate, angles and rivets are to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—These boxes will be single-riveted throughout with ⅜-in. diameter rivets, spaced 1¼ in. center to center.

**Caulking and Testing.**—All plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps. These boxes will be caulked on the outside, tested when full of water, and made absolutely tight and dry.

**Openings.**—Riveted to these boxes at approved locations, there will be furnished the necessary pipe flanges, which are to be of forged steel, taper-threaded.

**Painting.**—After completion, these boxes will be painted with one coat of graphite paint on the outside.

### One 500-bbl. Agitator

**Size and Style.**—This agitator will be of the vertical type, closed top, fifteen feet in diameter by twenty-five feet high (15 ft. by 25 ft.).

**General Construction.**—The shell of this agitator will be made in four courses of about seventy-five inches (75 in.) wide each, center to center of rivets. Located in the second ring and extending vertically about 60 in., there will be a cone, with its outer edge riveted to the shell of the agitator, and finishing at the center with a circular cap piece of about 24 in. diameter. In the skirt of the agitator, or that portion beneath the cone, there will be cut a doorway with a surrounding 2½-in. by 2½-in. by 5/16-in. angle frame. Around the top of the agitator, on the outside, there will be riveted a ring of 2-in. by 2-in. by ¼-in. steel angle; at the base on the outside will be an angle ring of 3 in. by 3 in. by ⅜ in.

**Steel Plate.**—The cone of the agitator will be made of ⅜-in. flange steel; the first three rings are to be of 5/16-in. tank steel, the balance of ¼-in. tank steel. All of the steel plate used in this agitator is to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—The vertical seams of the shell will be single-riveted with ⅝-in. diameter rivets, 2-in. pitch; the girth seams of the first three rings with single ⅝-in. rivets, 2-in. pitch. Where the cone is to be riveted to the shell of the agitator there will be a row of double ⅝-in. diameter rivets, spaced 2¾ in. center to center.

**Openings.**—This agitator will be supplied with a 4-in. cast-iron nozzle with companion flange, riveted to the cap piece at the base of the cone.

**Roof.**—This agitator is to be provided with a glove roof of No. 10 steel. Three flapper manholes are provided in the roof, equally spaced around the circumference, about 12 in. in from the edge of the tank. A balcony, with a wood floor supported by six angle iron brackets and angle railing, is provided, also a circular steel stairway.

**General.**—This agitator is not to be provided with a lead lining. It is to be erected complete on foundations, tested when full of water, and painted with one coat of approved paint on the outside. No guarantee is made that the agitator will remain absolutely tight for constant or intermittent use in the treatment of oils with sulphuric acid.

## Six Tanks, 6 ft. by 6 ft., Open Top

These tanks are to be of the vertical, cylindrical type, 6 ft. in diameter by 6 ft. high, open top, and are to be made of 3/16-in. steel plate throughout; the top angle is to be 2 in. by 2 in. by ¼ in. There is to be no angle iron in the bottom, which is to be flanged.

These tanks are to be riveted with ⅜-in. diameter rivets throughout, single-riveted and spaced 1¼ in. center to center. They are to be provided with the necessary flanges.

All are to be built complete in the shop, tested when full of water,

made absolutely tight and dry, given one coat of paint on the outside, and shipped intact.

Two Tanks, 10 ft. by 6 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, ten feet in diameter by six feet high (10 ft. by 6 ft.), each having a nominal capacity of about 80 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using two sketch plates, trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in one ring or course, 6 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:
Bottom sketch plates of $\frac{3}{16}$-in. plate, weighing 7.65 lb. per square foot.
First ring of shell of $\frac{3}{16}$-in. plate, weighing 7.65 lb. per square foot.

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2-in. by 2-in. by $\frac{1}{4}$-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2-in. by 2-in. by $\frac{1}{4}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell of these tanks will be bevel-sheared and caulked on the outside, and the bottom plates bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of these tanks are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and, before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive, and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—Each tank will also be provided with the following forged steel-pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where purchaser directs:

Two threaded for 2-in. pipe, taper-threaded, unless otherwise noted.

**Roof.**—These tanks are not to be provided with roofs.

**Riveting.**—The bottom of each tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅝-in. diameter rivets.

All girth seams are to be single, while all vertical seams are to be double-riveted, spaced 1¼ in. center to center.

**General.**—In general, these tanks are to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the field and in the shop.

## Four Tanks, 9 ft. by 8 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, nine feet in diameter by eight feet high (9 ft. by 8 ft.), each having a nominal capacity of about 100 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using two sketch plates, trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in one ring or course, 8 ft. wide.

**Material.**—These tanks will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

Bottom sketch plates of 3/16-in. plate, weighing 7.65 lb. per square foot.
First ring of shell of 3/16-in. plate, weighing 7.65 lb. per square foot.

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2-in. by 2-in. by ¼-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on inside of shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2-in. by 2-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shells of these tanks will be bevel-sheared and caulked on the outside, and the bottom plates bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—Each tank will be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

Two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—These tanks are not to be provided with roofs.

**Riveting.**—The bottom of each tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom with ⅝-in. diameter rivets and single-riveted to the shell with ⅝-in. diameter rivets. All girth seams are to be single and all vertical seams single-riveted, spaced 1¼ in. center to center.

**General.**—In general, these tanks are to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Three Tanks, 15 ft. by 10 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, fifteen feet in diameter by ten feet high (15 ft. by 10 ft.), each having a nominal capacity of about 300 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 20 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each about 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

Bottom rect. plates of 3/16-in. plate, weighing 7.32 lb. per square foot.
Bottom sketch plates of 3/16-in. plate, weighing 7.32 lb. per square foot.
First ring of shell of 3/16-in. plate, weighing 7.32 lb. per square foot.
Second ring of shell of 3/16-in. plate, weighing 7.32 lb. per square foot.

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle, to which the

shell and bottom will each be riveted; the bottom angle is to be on inside of shell. Around the top of the shell of each tank, on the outside, there will be an angle ring of 2-in. by 2-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell of these tanks will be bevel-sheared and caulked on the outside, and the bottom plates bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and, before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned. Upon completion, the shell is to be tested when full of water and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

Two threaded for 3-in. pipe; two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—These tanks are not to be provided with roofs.

**Riveting.**—The bottom of each tank is to be single-riveted with ⅜-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅜-in. diameter rivets. The ⅜-in. diameter rivets are to be spaced 1¼ in., center to center.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## One Tank, 15 ft. by 10 ft., Cone Roof

**Size and Style.**—This tank will be of the vertical, cylindrical type, fifteen feet in diameter by ten feet high (15 ft. by 10 ft.), having a nominal capacity of about 300 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of this tank will be made by using as many rectangular plates as practical, about 5 ft. by 20 ft., finishing

at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each about 5 ft. wide.

**Material.**—This tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:

Bottom rect. plates of 3/16-in. plate, weighing 7.32 lb. per square foot.
Bottom sketch plates of 3/16-in. plate, weighing 7.32 lb. per square foot.
First ring of shell of 3/16-in. plate, weighing 7.32 lb. per square foot.
Second ring of shell of 3/16-in. plate, weighing 7.32 lb. per square foot.

**Angles.**—The shell of this tank will be joined to the bottom by means of an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of shell. Around the top of the shell of the tank on the outside, there will be an angle ring of 2-in. by 2-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of this tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of this tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, this tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of this tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where purchaser directs:

Two threaded for 3-in. pipe; two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—This tank is to be provided with a steel roof of the conical type of No. 10 steel plate, supported by a pipe column and supplied with a 20-in. diameter wrought-steel hinged manhole.

**Riveting.**—The bottom of this tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅝-in. diameter rivets. The ⅝-in. diameter rivets are to be spaced 1¼ in., center to center.

**General.**—In general, this tank is to be built in a first-class workmanlike manner throughout; and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Fifteen Tanks, 20 ft. by 10 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, twenty feet in diameter by ten feet high (20 ft. by 10 ft.), each having a nominal capacity of about 500 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 20 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | |
|---|---|
| Bottom rect. plates of | No. 7 plate, weighing 7.32 lb. per square foot. |
| Bottom sketch plates of | No. 7 plate, weighing 7.32 lb. per square foot. |
| First ring of shell of | No. 7 plate, weighing 7.32 lb. per square foot. |
| Second ring of shell of | No. 7 plate, weighing 7.32 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and

dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

Two threaded for 3-in. pipe; two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—Each tank is to be provided with a wooden roof made of timbers and rafters of standard construction. The roof proper is to be of what is known commercially as 1-in. sheathing boards, one layer of builder's felt, and a top cover of No. 24 steel sheets. The sheets are to be painted on the under side before laying. The roof is to be provided with one hatchway of standard dimensions.

**Riveting.**—The bottom of each tank is to be single-riveted with $\frac{3}{8}$-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with $\frac{3}{8}$-in. diameter rivets. All vertical seams are to be single, all girth seams single-riveted. The spacing of the rivets is to be $1\frac{1}{4}$ in., center to center.

**Ladder.**—A steel ladder of approved design is to be supplied, running full height of each tank on the outside, neatly fitted opposite the manhole opening in roof.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Nine 3,000-bbl. Tanks

**Size and Style.**—These tanks will be of the vertical, cylindrical type, thirty feet in diameter by twenty-five feet high (30 ft. by 25 ft.), each having a nominal capacity of about 3,000 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 20 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in five rings or courses, each 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | |
|---|---|
| Bottom rect. plates of | No. 7 plate, weighing 7.32 lb. per square foot. |
| Bottom sketch plates of | No. 7 plate, weighing 7.32 lb. per square foot. |
| First ring of shell of | No. 5 plate, weighing 8.75 lb. per square foot. |
| Second ring of shell of | No. 6 plate, weighing 8.25 lb. per square foot. |
| Third ring of shell of | No. 7 plate, weighing 7.32 lb. per square foot. |
| Fourth ring of shell of | No. 7 plate, weighing 7.32 lb. per square foot. |
| Fifth ring of shell of | No. 7 plate, weighing 7.32 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank on the outside there will be an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where purchaser directs:

Two threaded for 3-in. pipe; two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—Each tank is to be provided with a wooden roof made of

timbers and rafters of standard construction. The roof proper is to be of what is known commercially as 1-in. sheathing boards, one layer of builder's felt, and a top cover of No. 24 steel sheets. The sheets are to be painted on the under side before laying. This roof is to be provided with one hatchway of standard dimensions.

**Riveting.**—The bottom of each tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅝-in. diameter rivets. All vertical seams are to be single and all girth seams double-riveted. Spacing of the rivets is to be 1¼ in., center to center.

**Ladder.**—A steel ladder of approved design is to be supplied, running the full height of each tank on the outside, neatly fitted opposite the manhole opening in the roof.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

### One Acid Tank, 10 ft. by 10 ft.

This tank is to be of the vertical cylindrical type with globe roof and flat bottom. It is to be made throughout of ⅜-in. steel plate. The shell plates are to run vertically.

There is to be no angle iron used in its construction, and it is to be designed to eliminate as many rivet seams as is practicable.

It is to be provided with a tar plug and stuffing box; the tar plug is to have a ground seat and is to be operated with a lever. The tank is to be provided with additional flanges to suit the purchaser's requirements, and it is to be riveted throughout with ⅝-in. diameter rivets.

This tank is to be built complete in the shop, tested when full of water, given one coat of paint on the outside, and shipped intact.

### Two Bleachers, 30 ft. by 8 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, thirty feet in diameter by eight feet high (30 ft. by 8 ft.), each having a nominal capacity of about 1,000 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 20 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in one ring or course, eight feet (8 ft.) wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

Bottom rect. plates of $\frac{3}{16}$-in. plate, weighing 7.32 lb. per square foot.
Bottom sketch plates of $\frac{3}{16}$-in. plate, weighing 7.32 lb. per square foot.
First ring of shell of $\frac{3}{16}$-in. plate, weighing 7.32 lb. per square foot.

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 2½-in. by 2½-in. by $\frac{5}{16}$-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be kept on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of a 2½-in. by 2½-in. by $\frac{5}{16}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where purchaser directs:

Two threaded for 3-in. pipe; and two threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—Each tank is to be provided with a wooden roof made of timbers and rafters. The roof proper is to be of what is known commercially as 1-in. sheathing boards covered with one layer of builder's felt and a top cover of No. 24 steel sheets. The under side of the steel sheets is to be given one coat of paint before laying. The roof is to be provided with four hatchways, about 30 in. by 72 in.

**Riveting.**—The bottom of each tank is to be single-riveted with $\frac{3}{8}$-in. diameter rivets. The bottom angle is to be riveted to the bottom and single-riveted to the shell with $\frac{3}{8}$-in. diameter rivets. All girth

seams and all vertical seams are to be single. The spacing of the rivets is to be 1¼ in., center to center.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Two Blow Cases

These drums, or blow cases, are to be of the horizontal cylindrical style, thirty inches in diameter by forty-eight inches deep (30 in. by 48 in.). They are to be made throughout of 5/16-in. steel plate. The heads are to be dished.

These cases are to be provided with two 2-in. wrought steel flanges, or their equivalent, to be placed where required. They are to be built complete in the shop, tested when full of water, made absolutely tight and dry, and given one coat of paint on the outside.

## Ten Filters, 7 ft. by 12 ft.

**Size and Style.**—These filters will be of the vertical cylindrical type, closed top and bottom, seven feet in diameter by twelve feet high (7 ft. by 12 ft.).

**General Construction.**—The shell of these filters will be made in three equal rings, two plates to the ring. The heads are to be made of one solid plate each, dished and flanged. The dishing is to be made on the 9-ft. radius.

**Steel Plate.**—The shell of these filters is to be of ¼-in. steel plate, and the heads of 5/16-in. All steel plate used in these filters is to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—These filters are to be riveted throughout with 5/8-in. diameter rivets. The girth seams are to be single, the vertical seams double-riveted.

**Openings.**—In the top heads of these filters is to be placed one 18-in. diameter cast-iron manhole, with bolted cover. In the bottom heads of the filters is to be placed one 6-in. cast-iron nozzle, tapped on both sides for 2-in. pipe; the nozzle is to be fitted with a blank head tapped for ½-in. pipe.

In the lower ring of the filter, just above the lower head, is to be located one 16-in. by 18-in. cast-iron rectangular manhole with a hinged cover; the cover is to be made with a threaded outlet for 5-in. pipe, which is to be specially designed, so that the contents can be easily discharged from the door. There is also to be located one 2-in. side-hill flange on the top head of the filters.

**Brackets.**—There are to be provided four heavy cast-iron brackets, 8 ft. by 8 ft. by 8 ft., and two are to be placed in the upper ring and two at the lower ring; they are to be placed opposite each other.

**Filter Plate.**—The bottom is to be designed to support a filter plate of five sections made of ¼-in. steel plate, perforated with ⅜-in. holes, spaced 1 in. apart. It is to be supported by an angle ring placed near the conjunction of the lower head with the shell. There are to be two angles placed crosswise, as well as a 2-in. by 2-in. by ¼-in. vertical support for each cross angle.

**Extra Angle.**—About 6 in. down from the top head of the shell of the filters, on the outside, is to be placed an angle ring, 2 in. by 2 in. by ¼ in.

**General.**—These filters are to be built complete in the shop, tested when full of water, made absolutely tight and dry, given one coat of paint on the outside, and shipped intact.

## 2. Specifications for a Refinery at Chanute, Kansas

### Eight Horizontal Crude Stills

**Size and Style.**—These stills will be of the horizontal type, ten feet in diameter by forty feet long (10 ft. by 40 ft.), in the shell.

**General Construction.**—The bottom of each still will be made in one solid plate, one hundred eight inches wide (108 in.) and forty feet (40 ft.) long, running the full length of the shell.

The upper part of the shell will be made in seven courses, running girthwise, one sheet to each course or ring. Each of the mainheads, or ends, will be made in two plates, with a double row of rivets through the center, and will be dished outward and flanged-in.

**Dome.**—On the center course of each still there will be a dome, thirty-six inches in diameter by thirty-six inches high (36 in. by 36 in.). The shell and head of the dome are each to be made in one plate; the dome head is to be dished upward and flanged-in.

**Steel Plate.**—Each still will be built throughout of the best quality open-hearth homogeneous flange steel, with the exception of the one long bottom plate, which is to be made of the best still-bottom steel. All the steel entering into the construction of this still shall conform to the standard specifications adopted by the Association of American Steel Manufacturers.

| | | |
|---|---|---|
| Bottom | of still to be of | ½-in. steel plate. |
| Shell | of still to be of | ⅜-in. steel plate. |
| Heads | of still to be of | ½-in. steel plate. |
| Dome | of still to be of | 5/16-in. steel plate. |
| Dome head | to be of | ⅜-in. steel plate. |

**Riveting.**—Each still will be double-riveted throughout with ¾-in. diameter rivets, excepting the dome head, manhole neck, flanges, etc., which will be single-riveted. All rivets must completely fill the holes and have full heads concentric with the body of the rivet, of a height not less than ½ in.

The diameter of the punch shall not exceed the diameter of the rivets to be used by more than 1/16 in., and all holes must be clean-cut without torn or ragged edges. Rivet holes must be accurately spaced, the use of drift pins being allowed only for the purpose of bringing members together, and must not be driven with such force as to disturb the metal about the holes.

All rivets are to be of first-class quality, made to conform to the standard specifications adopted by the Association of American Steel Manufacturers, and spaced in accordance with the best known rules for this class of work.

**Openings.**—On the top of each still, at an approved location, there will be two 20-in. diameter cast-iron still manholes, complete with cover, crossbar and screw. On the front head of the still, near the bottom, will be a 20-in. diameter steel manhole neck, finishing at the outer end with a wrought iron angle ring and bolted plate steel cover secured with 3/4-in. diameter square-shouldered bolts; a wrought-iron crane for carrying the cover is to be furnished and attached to the manhole neck. The neck is to be made of 1/2-in. steel plate.

Each still will be provided with forged steel pipe flanges, all to be taper-threaded, for standard pipe connections to meet the requirements. If preferred, there will be furnished, instead of the 3-in. flange in the back end of the bottom, one 3-in. diameter cast-iron tar plug with cast-iron outlet nozzle having a wrought-iron rod extending up through the shell of the still in line with the outlet nozzle beneath; to the bottom of this tar plug outlet nozzle there will be bolted one cast-iron flange, taper-threaded for 3-in. pipe. The tar plug is to be operated by a wheel handle or lever. The tar plug and seat are to be ground joint.

**Castings.**—For supporting each still on masonry there will be riveted to the shell of the still fourteen heavy cast-iron or pressed steel brackets, seven on each side. These brackets are to be riveted to the shell of the still with 1-in. diameter rivets. For each lug a channel, 12 in. by 36 in., with two rollers, properly fitted, is to be included.

**Caulking and Testing.**—All plates are to be neatly and carefully bevel-sheared on a rotary beveller for outside and inside caulking, and the corners are to be scarfed so as to insure tight joints at the laps. Each still is to be thoroughly caulked on the outside and inside, and made absolutely tight and dry.

## Four Horizontal Steam Stills

**Size and Style.**—These stills will be of the horizontal type, ten feet in diameter by forty feet long (10 ft. by 40 ft.).

**General Construction.**—The shell of each still will be made in seven roundabout courses, of equal width. Each end of the still will be made in two plates with a double row of rivets through the center, and will

be dished outward and flanged-in. On top of the shell will be a dome, 3 ft. in diameter by 6 ft. high (See Fig. 248).

**Steel Plate.**—Each still will be built throughout of open-hearth homogeneous steel, conforming to the standard specifications adopted by the Association of American Steel Manufacturers.

Shell of still made of 5/16-in. steel plate.
Ends or heads made of 5/16-in. steel plate.
Shell of dome made of 1/4-in. steel plate.
Head of dome made of 5/16-in. steel plate.

**Riveting.**—Each still will be riveted throughout with 5/8-in. diameter rivets. Longitudinal seams are to be double-riveted, girth seams single-riveted.

**Caulking and Testing.**—All plates are to be neatly and carefully sheared for outside caulking, and corners are to be scarfed so as to insure tight joints at the laps.

**Openings.**—In the top of the shell of each still, there will be one 20-in. diameter cast-iron manhole, complete with cover, crossbar and screw. In the front head of each still there will also be one 20-in. diameter steel manhole neck with steel angle ring and plate steel cover.

Riveted to the shell of each still in an approved location there will be seven 3-in. flanges, and to the dome one 8-in. flange; these flanges are to be of forged steel.

**Brackets.**—For supporting each still on masonry, there will be riveted to the shell fourteen cast-iron or pressed steel brackets, seven on each side. Each lug is to be provided with one steel plate, 12 in. wide by 24 in. long by 5/8 in. thick.

## One Condenser, 12 ft. by 8 ft. by 5 ft.

**Size and Style.**—This condenser will be rectangular in shape, twelve feet long, eight feet wide, by five feet deep (12 ft. by 8 ft. by 5 ft.), open top.

**General Construction.**—The bottom of this condenser will be constructed in two plates, each 5 ft. wide. Each plate is to be bent at the lower edge to a radius of 6 in., so as to form the sides of the condenser. Each end of the condenser will be made in one solid plate and flanged-in, and each side in one solid plate.

**Angles and Bracing.**—For the purpose of stiffening this condenser, there will be riveted around the top, on the outside, an angle band of 2½-in. by 2½-in. by 5/16-in. steel angle.

Riveted to each side of the condenser, there will be two vertical stiffeners of 2½-in. by 2½-in. by 5/16-in. steel angle, set opposite each other. Riveted to each end of the condenser, there will be two vertical angles of the same size. Running crosswise on top of the condenser, straight

and diagonally, there will be a set of braces of 2-in. by ¼-in. flat steel bar, bolted at each end to the top angle iron.

**Steel Plate.**—The steel plate used in the shell, that is, the sides and bottom, of this condenser will be of the best quality ¼-in. steel, and that used in the heads, or ends, of the best quality open-hearth homogeneous flange steel, ¼-in. thick. All the steel used will conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—This condenser will be single-riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center.

**Caulking and Testing.**—All the plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps.

This condenser is to be thoroughly caulked on the outside, tested when full of water, and made absolutely tight and dry.

**Openings.**—Riveted to the condenser at approved locations, there will be the necessary pipe flanges. All flanges are to be of forged steel, taper-threaded.

**Painting.**—After completion, this condenser will be given one coat of good red mineral paint on the outside.

## One Three-Compartment Condenser

**Size and Style.**—This condenser will be rectangular in shape, forty feet long, twenty-four feet wide, and six feet deep (40 ft. by 24 ft. by 6 ft.), open top, with two partitions lengthwise, making three (3) compartments, 40 ft. by 8 ft. by 6 ft., on one bottom.

**General Construction.**—The bottom of this condenser will be constructed in five courses, lengthwise, each 5 ft. wide across the bottom. Each course is to be made in three plates, bent at each lower edge to a radius of 6 in., so as to form the ends of the condenser, the two outer courses being each bent up to form the sides.

Each side and each partition will be made in three plates, and the partitions are to be flanged. Each end is to be made in two plates, flanged-in.

**Angles and Bracing.**—There will be riveted around the top, on the outside, and on top of each partition, an angle ring, or band, of 2½-in. by 2½-in. by 5/16-in. steel angle.

There will be riveted seven vertical angles, 3 in. by 3 in. by ⅜ in., to each side, seven to each partition, and three to each end.

Running crosswise of the condenser, straight and diagonally, there will be a set of braces of 2-in. by ¼-in. flat steel bar, bolted at each end to the top angle irons and to the partitions where they cross.

**Steel Plate.**—The steel plate used in the shell, that is, the sides and bottom, of this condenser will be of the best quality ¼-in. tank steel,

and that used in the heads, or ends, of the best quality open-hearth homogeneous flange steel, ¼ in. thick.

All the steel used is to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—This condenser will be single-riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center.

**Caulking and Testing.**—All the plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps.

The condenser is to be thoroughly caulked on the outside, tested when full of water, and made absolutely tight and dry.

**Openings.**—Riveted to the condenser at approved locations, there will be the necessary pipe flanges. All flanges are to be of forged steel, straight-threaded.

**Painting.**—After completion, this condenser will receive one coat of good red mineral paint on the outside.

## One Four-Compartment Condenser

**Size and Style.**—This condenser will be rectangular in shape, forty feet long, thirty-two feet wide and eight feet deep (40 ft. by 32 ft. by 8 ft.), open top, with three partitions lengthwise, making four compartments, 40 ft. by 8 ft. by 8 ft., on one bottom.

**General Construction.**—The bottom of this condenser will be constructed in seven courses lengthwise, each 5 ft. wide across the bottom. Each course is to be made in three plates, bent at each lower edge to a radius of 6 in., so as to form the ends of the condenser, the two outer courses being each bent up to form the sides. Each end is to be made in two plates, flanged-in.

Each side and each partition will be made in three plates; all partitions are to be flanged.

**Angles and Bracing.**—There will be riveted around the top, on the outside, and on top of each partition, an angle ring, or band, of 2½-in. by 2½-in. by 5/16-in. steel angle.

There will be riveted seven vertical angles, 3 in. by 3 in. by ⅜ in., to each side, seven to each partition, and three to each end.

Running crosswise of the condenser, straight and diagonally, there will be a set of braces of 2-in. by ¼-in. flat steel bar, bolted at each end to the top angle irons and to the partitions where they cross.

**Steel Plate.**—The steel plate used in the shell, that is, the sides and bottom, of this condenser, will be of the best quality ¼-in. tank steel, and that used in the heads, or ends, of the best quality open-hearth homogeneous flange steel, ¼ in. thick.

All the steel used is to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—This condenser will be single-riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center.

**Caulking and Testing.**—All the plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps.

The condenser is to be thoroughly caulked on the outside, tested when full of water, and made absolutely tight and dry.

**Openings.**—Riveted to the condenser at approved locations, there will be the necessary pipe flanges. All flanges are to be of forged steel, straight-threaded.

**Painting.**—After completion, this condenser will be given one coat of good red mineral paint on the outside.

## One Five-Compartment Condenser

**Size and Style.**—This condenser will be rectangular in shape, fifty feet long, thirty feet wide and six feet deep (50 ft. by 30 ft. by 6 ft.), open top, with four partitions lengthwise, making five compartments, 50 ft. by 6 ft. by 6 ft., on one bottom.

**General Construction.**—The bottom of this condenser will be constructed in six courses running lengthwise, each about 5 ft. wide. Each course is to be made in three plates bent at each lower edge to a radius of 6 in., so as to form the ends of the condenser, the two outer courses each being bent up to form the sides.

Each end of the condenser will be made in two plates, flanged-in. Each side and each partition will be made in four plates and to lap on the bottom plates; the partitions are to be flanged. This construction reduces to a minimum the number of cross seams on the bottom of condenser where the partitions are riveted.

**Angles and Bracing.**—Along the outside, on top of the condenser, and lengthwise, on top of each partition, is to be riveted a 2½-in. by 2½-in. by 5/16-in. steel angle; there will also be riveted to each end on the inside five vertical angles on to each side and to each partition eight vertical angles, all 2½ in. by 2½ in. by 5/16 in.

Running crosswise of the condenser, straight and diagonally, there will be furnished a set of braces of 2-in. by ¼-in. flat steel bar, punched for bolting at each end and, where they cross the partitions, to the top angle irons.

**Steel Plate.**—The steel plate used in the construction of this condenser will be of the best quality tank steel, ¼-in. thick, except that where flanging is required open-hearth flange steel is to be used.

All the steel used is to conform to the standard specifications adopted by the Association of American Steel Manufacturers.

**Riveting.**—This condenser will be single-riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center.

**Caulking and Testing.**—All the plates are to be neatly and carefully sheared for outside caulking, and the corners are to be scarfed so as to insure tight joints at the laps. The condenser is to be thoroughly caulked when full of water, and made perfectly tight and dry.

**Openings.**—There will be furnished the necessary pipe flanges to suit requirements. All flanges are to be forged steel, straight-threaded.

**Painting.**—After completion, this condenser is to receive one coat of red mineral paint.

## One Agitator, 18 ft. by 32 ft.

**Size and Style.**—This agitator will be of the vertical type, eighteen feet in diameter by thirty-two feet high (18 ft. by 32 ft.), in the shell. The cone is to have a 6-ft. drop. This agitator is to have a globe roof.

**General Construction.**—The shell of this agitator will be made in six equal courses. Located in the third ring and extending down vertically



FIG. 210.—General refinery work, consisting of one 1,200-bbl. agitator, one 2,400-bbl. agitator, three stills, 14 ft. 6 in. by 42 ft. in length, and three condenser tanks, 23 ft. by 48 ft. by 12 ft.

to a depth of about 6 ft., there will be a cone with the upper or outer edge riveted to the shell of the agitator; this is to finish at the bottom or apex with a cap piece of about 36 in. in diameter.

Around the top of the agitator, on the outside, there will be riveted an angle ring of 2½-in. by 2½-in. by $\frac{5}{16}$-in. steel angle, and at the bottom of the agitator, on the outside, there will be riveted an angle ring of 3-in. by 3-in. by $\frac{3}{8}$-in. steel angle.

**Supports.**—The cone is to be supported by means of four 4-in. pipe columns with flanges on the top and bottom.

**Steel Plate.**—The agitator will be made of the following thickness of plate:

| | |
|---|---|
| First four rings of shell | of 5/16-in. steel plate. |
| Balance of rings of shell | of 1/4-in. steel plate. |
| Cone | of 3/8-in. steel plate. |
| Roof | of No. 10 steel plate. |

All the steel plate used in the construction of this agitator is to conform to the specifications adopted by the Association of American Steel Manufacturers.

The plates used in the shell are to be of the best quality tank steel, while those used in the cone are to be of the best quality open-hearth flange steel.

**Riveting.**—This agitator is to be single-riveted throughout, excepting the cone roof, with 5/8-in. diameter rivets. The cone is to be double-riveted with 5/8-in. diameter rivets. The roof is to be single-riveted with 3/8-in. diameter rivets. All rivets in the cone and in the shell of the agitator above the cone are to be countersunk and driven flat from the inside.

**Roof.**—The roof is to be of the globe pattern, made of segmental plates of No. 10 steel plate capped with a circular plate of 3/16-in. plate, 36 in. in diameter, riveted securely on the outer edge to the top angle of the shell.

The roof is to be provided with four wrought steel flapper manholes.

**Gallery and Ladder.**—This agitator is to be provided with a gallery extending around the top at the proper distance; this is to be made of steel construction of angle bars and plates. There is to be a straight stairway running from the ground to the gallery.

**Caulking and Testing.**—All plates are to be bevel-sheared, but the agitator is not to be caulked or tested. However, the cone is to be caulked.

**Openings.**—At the apex of the cone, on a circular cap piece, will be furnished a cast-iron nozzle with a 6-in. standard pipe companion flange.

In the skirt of the agitator will be cut an opening, 30 in. wide, 60 in. high on the sides, with arched top, and stiffened around the edge with an angle iron, 2½ in. by 2½ in. by 5/16 in.

## One Tank, 9 ft. by 9 ft.

**Size and Style.**—This tank will be of the vertical, cylindrical type, nine feet in diameter by nine feet high (9 ft. by 9 ft.), having a nominal capacity of about 100 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of this tank will be made by using two sketch plates, trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in one ring or course, 9 ft. wide.

**Material.**—This tank will be built throughout of first-class quality open-hearth tank steel, and all the plates, angles and rivets are to be in

accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:

Bottom rect. plates of $\frac{3}{16}$-in. plate, weighing 7.65 lb. per square foot.
Bottom sketch plates of $\frac{3}{16}$-in. plate, weighing 7.65 lb. per square foot.

**Angles.**—The shell of this tank will be joined to the bottom by means of an angle ring of 2-in. by 2-in. by $\frac{1}{4}$-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2-in. by 2-in. by $\frac{1}{4}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell of this tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of this tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon the completion of the tank.

**Painting.**—After completion, this tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of this tank there is to be one cast-iron manhole, riveted on and provided with a cast-iron bolted cover complete.

**Flanges.**—This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

One threaded for 3-in. pipe; and one threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—This tank is not to be provided with a roof.

**Riveting.**—The bottom of this tank is to be single-riveted with $\frac{3}{8}$-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with $\frac{3}{8}$-in. diameter rivets. The shell of the tank is to be single-riveted throughout with $\frac{3}{8}$-in. diameter rivets, spaced $1\frac{1}{2}$ in. center to center.

**General.**—In general, this tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## One Tank, 20 ft. by 12 ft.

**Size and Style.**—This tank will be of the vertical, cylindrical type, twenty feet in diameter by twelve feet high (20 ft. by 12 ft.), having a nominal capacity of about 600 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of this tank will be made by using two rectangular plates and two sketch plates, trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each 6 ft. wide.

**Material.**—This tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:

| | |
|---|---|
| Bottom rect. plates of | 3/16-in. plate, weighing 7.65 lb. per square foot. |
| Bottom sketch plates of | 3/16-in. plate, weighing 7.65 lb. per square foot. |
| First ring of shell of | 3/16-in. plate, weighing 7.65 lb. per square foot. |
| Second ring of shell of | 3/16-in. plate, weighing 7.65 lb. per square foot. |
| Roof plates of | No. 10 plate, weighing 5.45 lb. per square foot. |

**Angles.**—The shell of this tank will be joined to the bottom by means of an angle ring of 2-in. by 2-in. by ¼-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2-in. by 2-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of this tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of this tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, this tank is to be given one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of this tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a

cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

One threaded for 3-in. pipe; one threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—This tank is to be provided with a globe roof of No. 10 steel, formed of segmental plates radiating from the center and riveted at the outer edge to the top angle iron on the shell of the tank. The roof is to finish at the center with a cap piece about 3 ft. in diameter, supported with a central pipe column of 3-in. pipe with flange top and bottom.

**Riveting.**—The bottom of this tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅝-in. diameter rivets. The shell of the tank is to be single-riveted throughout with ⅝-in. diameter rivets, spaced 1½ in. center to center. The roof is to be single-riveted with ⅝-in. rivets, 2-in. pitch.

**General.**—In general, this tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## One Tank, 20 ft. by 12 ft., Open Top

**Size and Style.**—This tank will be of the vertical, cylindrical type, twenty feet in diameter by twelve feet high (20 ft. by 12 ft.), having a nominal capacity of about 600 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of this tank will be made by using two rectangular plates and two sketch plates, trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each 6 ft. wide.

**Material.**—This tank will be built throughout of first-class open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:

| | | |
|---|---|---|
| Bottom rect. plates of | 3/16-in. plate, | weighing 7.65 lb. per square foot. |
| Bottom sketch plates of | 3/16-in. plate, | weighing 7.65 lb. per square foot. |
| First ring of shell of | 3/16-in. plate, | weighing 7.65 lb. per square foot. |
| Second ring of shell of | 3/16-in. plate, | weighing 7.65 lb. per square foot. |

**Angles.**—The shell of this tank will be joined to the bottom by means of an angle ring of 2-in. by 2-in. by ¼-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside,

there will be an angle ring of 2-in. by 2-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell of this tank will be bevel sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of this tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, this tank is to be given one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of this tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete. This tank will also be provided with the following forged steel pipe flanges, threaded for standard pipe connection, or their monetary equivalent, and located where the purchaser directs:

One threaded for 3-in. pipe; one threaded for 2-in. pipe, all flanges to be taper-threaded unless otherwise noted.

**Roof.**—This tank is not to be provided with a roof.

**Riveting.**—The bottom of this tank is to be single-riveted with ⅝-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with ⅝-in. diameter rivets. The shell of the tank is to be single-riveted throughout with ⅝-in. diameter rivets, spaced 1½ in. center to center.

**General.**—In general, this tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Four Tanks, 40 ft. by 12 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, forty feet in diameter by twelve feet high (40 ft. by 12 ft.), each having a nominal capacity of about 2,700 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 15 ft., finishing at

the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in three rings or courses, each about 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | | |
|---|---|---|
| Bottom rect. plates of | $\frac{13}{64}$-in. plate, weighing | 8.25 lb. per square foot. |
| Bottom sketch plates of | $\frac{13}{64}$-in. plate, weighing | 8.25 lb. per square foot. |
| First ring of shell of | $\frac{1}{4}$-in. plate, weighing | 10.2 lb. per square foot. |
| Second ring of shell of | $\frac{7}{32}$-in. plate, weighing | 8.75 lb. per square foot. |
| Third ring of shell of | $\frac{13}{64}$-in. plate, weighing | 8.25 lb. per square foot. |
| Roof plates of | $\frac{13}{64}$-in. plate, weighing | 8.25 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 3-in. by 3-in. by $\frac{5}{16}$-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 3-in. by 3-in. by $\frac{5}{16}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be given one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete.

**Roof.**—Each tank is to be provided with a water-top roof of No. 6 steel plate weighing 8.25 lb. The roof is to be supported by an intermediate frame of 6-in., 12¼-lb. I-beams and twenty-four radial sup-

ports of 4-in., 5¼-lb. channels; these are to be supported by one central pipe post, 6 in. in diameter, and six intermediate supports, 3 in. in diameter.

The water top is to be formed by extending the roof plates out onto the top angle of the shell and curbing with an angle, 5 in. by 3 in. by 5/16 in., riveted on top of the roof plates.

This roof is to be supplied with one 18-in. diameter manhole, made flush with top.

**Riveting.**—The bottom of each tank is to be single-riveted, and the bottom angle is to be riveted to the bottom and to the shell.

Each tank is to be riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center; the girth seams are to be single, the vertical seams double-riveted.

**Well.**—Riveted to the bottom of each tank is to be one well, 18 in. in diameter by 12 in. deep.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Two Tanks, 50 ft. by 12 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, fifty feet in diameter by twelve feet high (50 ft. by 12 ft.), each having a nominal capacity of about 4,000 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 15 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in three rings or courses, each about 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | | |
|---|---|---|
| Bottom rect. plates of | 13/64-in. plate, weighing | 8.25 lb. per square foot. |
| Bottom sketch plates of | 13/64-in. plate, weighing | 8.25 lb. per square foot. |
| First ring of shell of | ¼-in. plate, weighing | 10.2 lb. per square foot. |
| Second ring of shell of | 7/32-in. plate, weighing | 8.75 lb. per square foot. |
| Third ring of shell of | 13/64-in. plate, weighing | 8.25 lb. per square foot. |
| Roof plates of | 13/64-in. plate, weighing | 8.25 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 3-in. by 3-in. by 5/16-in. steel angle, to which the shell

and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 3-in. by 3-in. by $\frac{5}{16}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to receive one coat on the outside of a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete.

**Roof.**—Each tank is to be provided with a water-top roof of No. 6 steel plate weighing 8.25 lb. The roof is to be supported by an intermediate frame of 8-in., 18-lb. I-beams and thirty radial supports of 5-in., 8-lb. channels; these are to be supported by one central pipe post, 6 in. in diameter, and six intermediate supports, 4 in. in diameter.

The water top is to be formed by extending the roof plates out onto the top angle of shell and curbing with an angle, 5 in. by 3 in. by $\frac{5}{16}$ in., riveted on top of roof plates.

The roof is to be supplied with one 18-in. diameter manhole, made flush with top.

**Riveting.**—The bottom of each tank is to be single-riveted. The bottom angle is to be riveted to the bottom and to the shell.

Each tank is to be riveted throughout with $\frac{7}{16}$-in. diameter rivets, spaced $1\frac{1}{2}$ in. center to center; the girth seams are to be single and the vertical seams double-riveted.

**Well.**—Riveted to the bottom of each tank is to be one well, 18 in. in diameter by 12 in. deep.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## Two Tanks, 60 ft. by 15 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, sixty feet in diameter by fifteen feet high (60 ft. by 15 ft.), each having a nominal capacity of about 7,500 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 15 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in three rings or courses, each about 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | | | |
|---|---|---|---|
| Bottom rect. plates of | 13/64-in. | plate, weighing | 8.25 lb. per square foot. |
| Bottom sketch plates of | 13/64-in. | plate, weighing | 8.25 lb. per square foot. |
| First ring of shell of | 1/4-in. | plate, weighing | 10.2 lb. per square foot. |
| Second ring of shell of | 7/32-in. | plate, weighing | 8.75 lb. per square foot. |
| Third ring of shell of | 13/64-in. | plate, weighing | 8.25 lb. per square foot. |
| Roof of | No. 10 | plate, weighing | 5.45 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 3-in. by 3-in. by 3/8-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of each tank, on the outside, there will be an angle ring of 2½-in. by 2½-in. by 5/16-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete.

**Roof.**—Each tank is to be provided with a globe roof of No. 10 steel and is to be supported by ten trussed fans of 2½-in. by 2½-in. by 5/16-in. angle with 1-in. round iron with turn-buckles. Plates are to overlap each other, capped in the center with a circular plate, 60 in. in diameter, riveted to the outside top angle of the tank; and there is to be a central pipe column of 6-in. pipe, secured to top and bottom by means of a companion flange.

The roof is to be provided with a hinged manhole, 20 in. in diameter.

**Riveting.**—The bottom of each tank is to be single-riveted with 7/16-in. diameter rivets. The bottom angle is to be single-riveted to the bottom and to the shell with 7/16-in. diameter rivets.

Each tank, with the exception of the roof, is to be riveted with 7/16-in. diameter rivets; the girth seams are to be single and the vertical seams double-riveted. The roof to be riveted with 3/8-in. diameter rivets.

**Wells.**—Each tank is to have one suction well on the bottom, 18 in. in diameter by 12 in. deep.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

## One Tank, 60 ft. by 15 ft., Special

**Size and Style.**—This tank will be of the vertical, cylindrical type, sixty feet in diameter by fifteen feet high (60 ft. by 15 ft.), having a nominal capacity of about 7,500 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of this tank will be made by using as many rectangular plates as practical, about 5 ft. by 15 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in three rings or courses, each about 5 ft. wide.

**Material.**—This tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used in this tank:

| | | | |
|---|---|---|---|
| Bottom rect. plates of | 13/64-in. plate, | weighing | 8.25 lb. per square foot. |
| Bottom sketch plates of | 13/64-in. plate, | weighing | 8.25 lb. per square foot. |
| First ring of shell of | ¼-in. plate, | weighing | 10.2 lb. per square foot. |

| | | |
|---|---|---|
| Second ring of shell of | $\frac{7}{32}$-in. plate, weighing | 8.75 lb. per square foot. |
| Third ring of shell of | $\frac{13}{64}$-in. plate, weighing | 8.25 lb. per square foot. |
| Roof plates | No. 6 plate, weighing | 8.25 lb. per square foot. |

**Angles.**—The shell of this tank will be joined to the bottom by means of an angle ring of 3-in. by 3-in. by $\frac{3}{8}$-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 3-in. by 3-in. by $\frac{5}{16}$-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of this tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of this tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, this tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of this tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete.

**Roof.**—This tank is to be provided with a water top roof of No. 6 steel plates weighing 8.25 lb. The roof is to be supported by an intermediate frame of 8-in., 18 lb. I-beams and thirty-six supports of 6-in. channels; these are to be supported by one central pipe post, 6 in. in diameter, and six intermediate supports, 4 in. in diameter. The water top is to be formed by extending the roof plates out onto the top angle of the shell and curbing with an angle, 5 in. by 3 in. by $\frac{5}{16}$ in., riveted on top of roof plates.

The roof is to be supplied with one 18-in. diameter manhole, made flush with top.

**Riveting.**—The bottom of this tank is to be single-riveted, and the bottom angle is to be riveted to the bottom and shell.

This tank is to be riveted throughout with $\frac{7}{16}$-in. diameter rivets, spaced $1\frac{1}{2}$ in. center to center; the girth seams are to be single, the vertical seams double-riveted.

**Well.**—Riveted to the bottom of the tank is to be one well, 18 in. in diameter by 12 in. deep.

**General.**—In general, this tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

### One Acid Tank

**Size and Style.**—This tank will be of the horizontal, cylindrical type, seven feet in diameter by twenty-five feet long (7 ft. by 25 ft.), in the shell.

**Shell.**—The shell of this tank is to be constructed in five roundabout courses, two plates to each course.

**Heads.**—The ends, or main heads, of this tank will each be in one solid plate, dished outward and flanged-in.

**Material.**—All the steel plate and rivets entering into the construction of this tank will be of the best of their several kinds, conforming to the specifications adopted as standard by the Association of American Steel Plate Manufacturers. All shell plates are to be of $\frac{5}{16}$-in. tank steel; the main heads of $\frac{5}{16}$-in. flange steel.

**Riveting.**—The longitudinal seams are to be double-riveted and all girth seams single-riveted. The rivets throughout are to be $\frac{5}{8}$ in. in diameter.

**Caulking and Testing.**—This tank is to be thoroughly caulked, both inside and outside when full of water, and is to be made absolutely tight and dry.

**Openings.**—On the center of the shell, on the top, there will be one eleven in. by fifteen in. (11 in. by 15 in.) pressed steel manhole complete. On the main head near the bottom there will be one 4-in. cast-iron pipe flange with the thread parallel to the horizontal line of the tank. All flanges are to be taper-threaded.

**Painting.**—After completion, this tank is to be given one coat of graphite paint on the outside.

### Two Bleachers, 60 ft. by 10 ft.

**Size and Style.**—These tanks will be of the vertical, cylindrical type, sixty feet in diameter by ten feet high (60 ft. by 10 ft.), each having a nominal capacity of about 5,000 bbl., more or less, rated at 42 gal. per barrel.

**Bottom and Shell.**—The bottom of each tank will be made by using as many rectangular plates as practical, about 5 ft. by 15 ft., finishing at the outer edge with sketch plates trimmed to conform to the circumference of the bottom angle.

The shell will be constructed in two rings or courses, each about 5 ft. wide.

**Material.**—Each tank will be built throughout of first-class quality open-hearth tank steel, all the plates, angles and rivets to be in accordance with their respective specifications as adopted by the Association of American Steel Manufacturers.

Thicknesses of plate to be used:

| | |
|---|---|
| Bottom rect. plates of | No. 6 plate, weighing 8.25 lb. per square foot. |
| Bottom sketch plates of | No. 6 plate, weighing 8.25 lb. per square foot. |
| First ring of shell of | No. 6 plate, weighing 8.25 lb. per square foot. |
| Second ring of shell of | No. 6 plate, weighing 8.25 lb. per square foot. |
| Roof of | No. 10 plate, weighing 5.45 lb. per square foot. |

**Angles.**—The shell of each tank will be joined to the bottom by means of an angle ring of 3-in. by 3-in. by ⅜-in. steel angle, to which the shell and bottom will each be riveted; the bottom angle is to be on the inside of the shell. Around the top of the shell of the tank, on the outside, there will be an angle ring of 2½-in. by 2½-in. by ¼-in. steel angle. There will be no inside top angle unless expressly stipulated.

**Shearing and Caulking.**—The plates in the shell and roof of each tank will be bevel-sheared and caulked on the outside, and the bottom plates will be bevel-sheared and caulked on the inside; the angles will not be beveled. All caulking is to be done in a careful workmanlike manner.

**Testing.**—The bottom and first ring of each tank are to be fitted up on horses, the bottom and bottom angle riveted up and caulked; and before lowering to the permanent foundation, the bottom and bottom angle are to be tested by filling with 6 in. of water, and caulked tight and dry to the acceptance of a representative, which test shall be final and conclusive as far as the bottom and bottom angle are concerned.

Upon completion, the shell is to be tested when full of water, and caulked tight and dry in the presence of a representative, who is to accept same from the foreman in charge of the work by a written letter of acceptance to him. This inspection and acceptance is to be final and conclusive and is to be made promptly upon completion of the tank.

**Painting.**—After completion, each tank is to be painted one coat on the outside with a first-class quality graphite paint.

**Openings.**—In the first ring of the shell of each tank there is to be one cast-iron manhole, 18 in. in diameter, riveted on and provided with a cast-iron bolted cover complete.

**Roof.**—Each tank is to be provided with a globe roof of No. 10 steel plate, and is to be supported by ten steel trusses, supported by a central pipe column of 6-in. pipe.

The roof is to have a rise of 6 ft. in the center; the plates are to overlap each other, capped in the center with a 72-in. diameter plate of ¼-in. steel, and riveted to the outside top angle of the tank.

Each tank is to be provided with not more than six steel hatchways of standard dimensions.

**Riveting.**—The bottom of each tank is to be single-riveted, and the bottom angle is to be riveted to the bottom and shell.

Each tank is to be riveted throughout with 7/16-in. diameter rivets, spaced 1½ in. center to center, with the exception of the roof, which is to be riveted with ⅜-in. diameter rivets.

**General.**—In general, each tank is to be built in a first-class workmanlike manner throughout, and the work will be open at any and all reasonable times to inspection, both in the shop and field.

### 3. Specifications for Two Refineries in Oklahoma and One in Texas

The following specifications, which are published through the courtesy of The Reeves Brothers Company, of Alliance, Ohio, cover the materials used in the construction of recently erected refineries at Cushing and Ponca City, Oklahoma, and at Gainesville, Texas. Each of the first two has a refining capacity of 4,000 bbl. of crude petroleum per day, while the last-mentioned has subsequently been enlarged to a capacity of 13,500 bbl. per day.

#### Items Included

Two (2) tanks, 55,000-bbl. capacity, 115 ft. diameter by 30 ft. high.

One (1) tank, 95 ft. diameter by 30 ft. high.

Eleven (11) tanks, 50 ft. diameter by 30 ft. high.

Twelve (12) run tanks, 35 ft. diameter by 10 ft. high.

Three (3) bleacher tanks, 35 ft. diameter by 10 ft. high, with ten (10) doors.

Two (2) bleacher tanks, 35 ft. diameter by 10 ft. high, one (1) door.

One (1) acid tank, 10 ft. diameter by 10 ft. high.

One (1) caustic tank, 10 ft. diameter by 5 ft. high, open top.

One (1) caustic tank, 6 ft. diameter by 5 ft. high, open top.

Two (2) agitators, 25 ft. diameter by 35 ft. high.

One (1) condenser, 35 ft. by 16½ ft. by 8 ft. deep.

Seven (7) condensers, 33½ ft. by 25 ft. by 8 ft.

Six (6) condensers, 25 ft. by 8 ft. by 4 ft.

Seven (7) sets condenser supports for 33½ ft. by 25 ft. by 8 ft. condensers.

One (1) condenser support for 35 ft. by 16½ ft. by 8 ft. condenser.

Two (2) crude stills, 12 ft. diameter by 35 ft. long.

All the above to be shipped, completely K. D., customer to erect.

Twenty (20) stills, 8 ft. diameter by 20 ft. long.

Eight (8) stills, 8 ft. diameter by 8 ft. long.

Eight (8) drums, 30 in. diameter by 7 ft. high.

Three (3) blow cases, 36 in. diameter by 5 ft. long.

All the above material to be shipped completely knocked down, with the exception of the last four items, which are to be built up complete at plant, tested, made tight and shipped intact.

**Material.**—*Plates.*—All plates shall be in accordance with Manufacturers Standard Specifications for the different grades.

*Rivets.*—All rivets to be made of soft rivet steel in accordance with Manufacturers Standard Specifications.

*Angles, Bars and Forgings.*—Angles, bars and forgings to conform to Manufacturers Standard Specifications.

*Castings.*—All iron castings shall be good quality gray iron.

All steel castings good quality cast steel, free from blow holes.

**Workmanship.**—Workmanship to be first-class in every respect.

*Punching.*—All holes shall be accurately layed out and punched true, using punches 1/16 in. larger in diameter than the diameter of rivets 1/2 in. and larger, and 1/32 in. larger in diameter than the diameter of rivets 7/16 in. and smaller. Punches and dies shall be sharp and in first-class condition.

*Flanging.*—Flanging and dishing shall be well done and in accordance with best practice.

*Planing and Shearing.*—Caulking edges shall be neatly planed or rotary bevel-sheared, as called for in description below.

*Scarfing.*—Lapping corners shall be carefully and neatly scarfed and chipped where necessary.

*Rolling.*—Care shall be exercised in rolling, and plates must be true to required sweep.

*Fitting.*—Before shipment is made, the various parts shall be carefully fitted and corrections made that may be necessary.

## Description

### Two (2) Tanks, 115 ft. Diameter by 30 ft. High

**Material.**—Bottom rectangle, 8.29 lb. Bottom sketches, 9.56 lb. Bottom angle, 4 in. by 4 in. by 1/2 in. First ring, 21 lb. Second ring, 17.85 lb. Third ring, 15.3 lb. Fourth ring, 11.47 lb. Fifth ring, 10.2 lb. Sixth ring, 8.29 lb. Top angle, 3 in. by 3 in. by 3/8 in.

**Riveting.**—Bottom to be single-riveted, 7/16-in. rivets, 1½-in. pitch.
Bottom angle, single-riveted, 3/4-in. rivets, 2⅜-in. pitch.
First ring verticals, triple-riveted, 3/4-in. rivets, 3-in. pitch.
Second ring verticals, triple-riveted, 3/4-in. rivets, 3⅜-in. pitch.
Third ring verticals, double-riveted, 3/4-in. rivets, 2⅞-in. pitch.
Fourth ring verticals, double-riveted, 5/8-in. rivets, 2½-in. pitch.
Fifth ring verticals, double-riveted, 7/16-in. rivets, 1½-in. pitch.
Sixth ring verticals, double-riveted, 7/16-in. rivets, 1⅝-in. pitch.
Top angle, single-riveted, 7/16-in. rivets, 3-in. pitch.

All horizontal seams single-riveted.

**Fittings.**—Tank to be provided with one (1) 20-in. manhole in center of first ring with reinforcing plate and gasket.

Also the following flanges: One (1) 8-in. flange shrunk on and riveted on nipple for swing pipe. Two (2) 4 in. straight thread flanges, one (1) 5-in. straight thread, two (2) 2-in. straight thread flanges. The two (2) 2-in. straight thread to be equipped with goose neck or steam bends.

Tank to be provided with 370 No. 22 gauge 36-in. by 120-in. steel galvanized sheets with the necessary 2D galvanized large head roofing nails for nailing the steel sheets to the roof.

One steel stairs with cab to be provided, also one standard windlass, and four (4) 8-in. galvanized-iron gauge hatches.

The necessary rivets plus 10 per cent. to be furnished.

### One Tank, 95 ft. by 30 ft.

**Material.**—Bottom rectangle, 8.29 lb. Bottom sketches, 9.56 lb. Bottom angle, 4 in. by 4 in. by ½ in. First ring, 17.85 lb. Second ring, 15.3 lb. Third ring, 11.47 lb. Fourth ring, 10.2 lb. Fifth ring, 8.92 lb. Sixth ring, 8.29 lb. Top angle, 3 in. by 3 in. by ⅜ in.

**Riveting.**—Bottom, single-riveted, 7/16-in. rivets, 1½-in. pitch.
Bottom angle, single-riveted, ¾-in. rivets, 2⅜-in. pitch.
First ring verticals, double-riveted, ¾-in. rivets, 2¾-in. pitch.
Second ring verticals, double riveted, ¾-in. rivets, 2⅞-in. pitch.
Third ring verticals, double-riveted, ⅝-in. rivets, 2½-in. pitch.
Fourth ring verticals, double-riveted, 7/16-in. rivets, 1½-in. pitch.
Fifth ring verticals, double-riveted, 7/16-in. rivets, 1½-in. pitch.
Sixth ring verticals, double-riveted, 7/16-in. rivets, 1⅝-in. pitch.
Top angle, single-riveted, 7/16-in. rivets, 3-in. pitch.

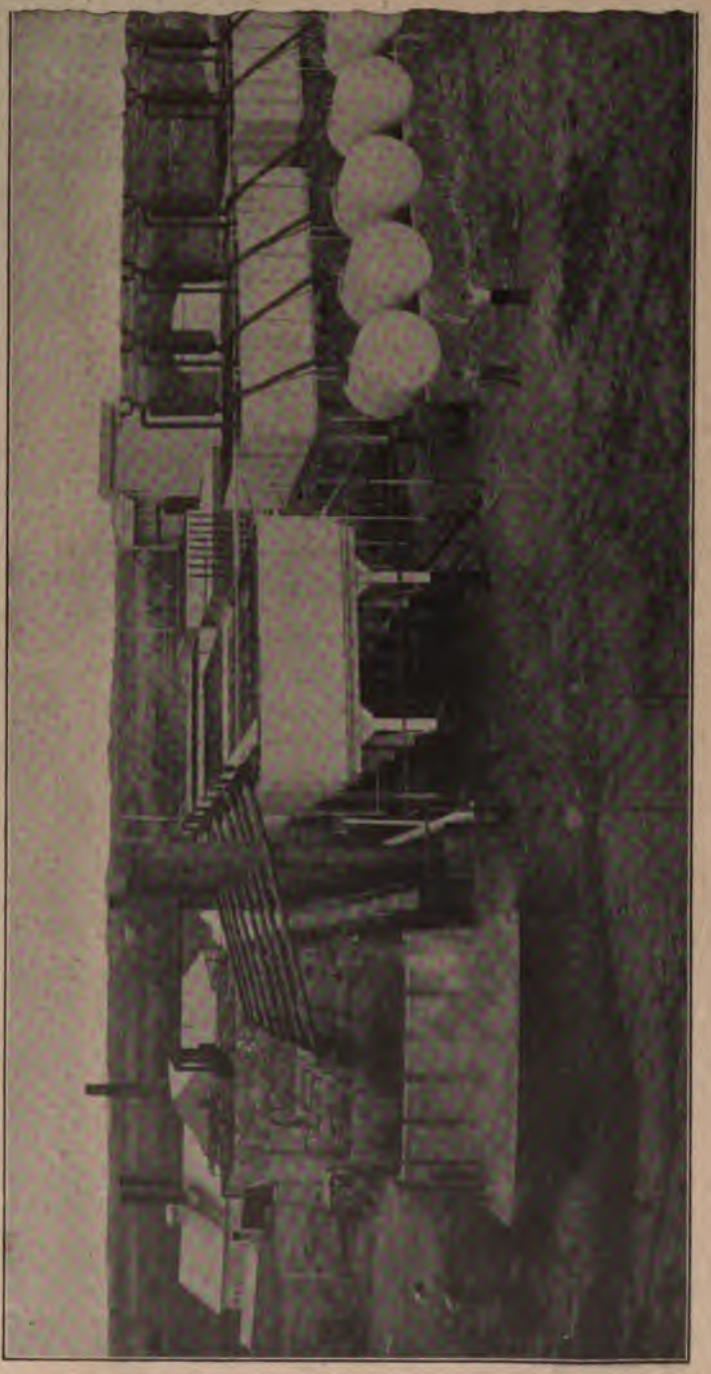
**Fittings.**—Tank to be provided with one (1) 6-in. flange shrunk on nipple for swing pipe. Two (2) 4-in. flanges, one (1) 5-in. flange and two (2) 2-in. flanges, all straight threads. Also two (2) 2-in. steam bends, standard steel stairs with cab, one (1) 20-in. manhead with gasket and reinforcing plate.

270 sheets No. 22 gauge, 36-in. by 120-in. steel galvanized sheets for roofing and necessary 2D large head roofing nails, and one windlass and four (4) 8-in. galvanized-iron gauge patches.

### Eleven (11) Tanks, 50 ft. Diameter by 30 ft. High

**Material.**—Bottom, 8.29 lb. plate. Bottom angle, 3 in. by 3 in. by ⅜ in. First ring, 11.47 lb. Second ring, 10.2 lb. Third ring, 8.92 lb. Fourth ring, 8.29 lb. Fifth ring, 8.29 lb. Sixth ring, 7.65 lb. Top angle, 2½ in. by 5/16 in.

**Riveting.**—Bottom, single-riveted, 7/16-in. rivets, 1½-in. pitch. Bottom angle, single-riveted, ⅝-in. rivets, 2-in. pitch. First ring verti-



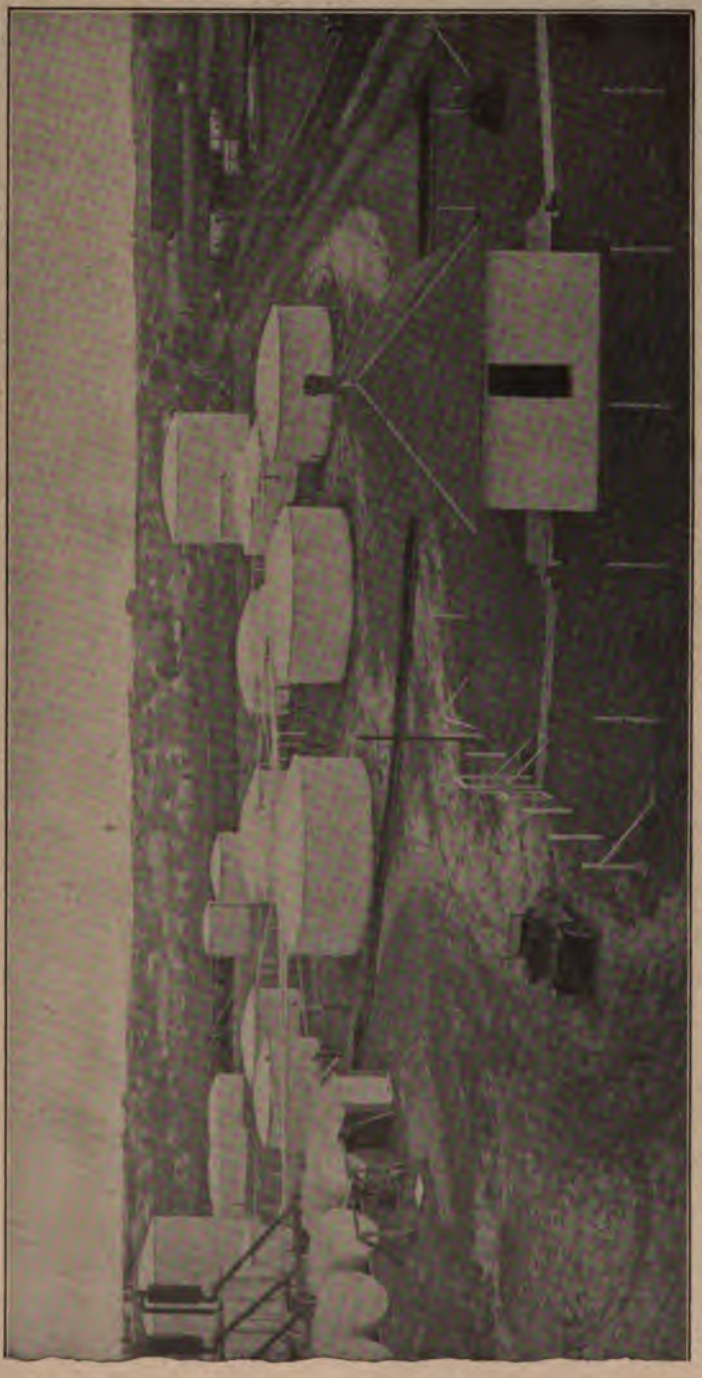


FIG. 211.—A Refinery at Cushing, Oklahoma, erected in accordance with the specifications on pages 683–97.

cals, double-riveted, 5/8-in. rivets, 2½-in. pitch. Second and third ring verticals, double-riveted, 7/16-in. rivets, 1½-in. pitch. Fourth, fifth and sixth ring verticals, single-riveted, 7/16-in. rivets, 1½-in. pitch. Top angle, single-riveted, 7/16-in. rivets, 3-in. pitch. All horizontal seams single-riveted throughout.

**Fittings.**—Tanks to be provided with one (1) 20-in. manhole; one (1) 6-in., one (1) 4-in. and one (1) 2-in. flange in first ring of tank, and two (2) 2-in. steam flanges.

Tank to be provided with standard ladder on the outside.

**Roof.**—Roof to be umbrella type with a rise of 5 ft. in the center. Plates to be No. 10 gauge or 5.45 lb. Single-riveted, 3/8-in. rivets, 1½-in. pitch. Roof plates to be sheared for caulking and to be provided with one (1) 6-in. pipe post in the center and one (1) steel hatch, 18-in. square, near the edge. Necessary rivets plus 10 per cent. to be furnished.

### Twelve (12) Tanks, 35 ft. Diameter by 10 ft. High

**Material.**—Bottom, 7.32 lb. Bottom angle, 3 in. by 3 in. by 3/8 in. First ring, 8.29 lb. Second ring, 7.32 lb. Roof, 5.45 lb.

**Riveting.**—Tank to be single-riveted throughout with 7/16-in. rivets, 1½-in. pitch, with the exception of the roof, which will have 3/8-in. rivets, 1½-in. pitch.

**Roof.**—Roof to be provided with one (1) 4-in. pipe column in the center.

Tank to be provided with one (1) 20-in. manhead; one 6-in., one 4-in. and one 2-in. flange, also one 18-in. square roof hatch and standard steel ladder on outside of tank.

### Three (3) Bleacher Tanks, 35 ft. in Diameter by 10 ft. High

**Construction.**—To be the same construction as the 35-ft. by 10-ft. run tanks, with the exception that same will be provided with ten doors, 24 in. by 36 in., on the roof.

### Two (2) Bleacher Tanks, 35 ft. in Diameter by 10 ft. High

**Construction.**—To be the same construction as the 35-ft. by 10-ft. run tanks, with the exception of one door, 24 in. by 36 in., and one 5-in., one 4-in., and one 2-in. flange.

### One Acid Tank, 10 ft. Diameter by 10 ft. High

**Dimensions.**—Acid tank to be 10 ft. in diameter by 10 ft. high in the shell.

**Material.**—Bottom made up of two plates, 3/8-in. or 15.3-lb., flanged up for connection to shell.

Shell of acid tank to be made up of two rings, 3/8-in. or 15.3-lb. plate.

Roof shall be made of two plates No. 8 B.W.G. Top angle to be 2½ in. by 2½ in. by 5/16 in.

**Manhead.**—One 16-in. diameter manhead with loose cover shall be provided with roof.

**Flanges.**—Flanges, plug, rod, stuffing box, gland, levers, brackets, etc., will be furnished.

**Riveting.**—Bottom cross seam, girth and verticals in acid tank shall all be single-riveted, ¾-in. rivets, 2.4-in. pitch. Top angle shall be riveted to top ring of acid tank, ¾-in. rivets, 4-in. pitch. Roof to top angle, ¾-in. rivets, 4-in. pitch. Roof seams, ⅜-in. rivets, 1½-in. pitch.

### One (1) Caustic Tank, 10 ft. in Diameter by 5 ft. High, Open Top

**Shell.**—Shell to be made of ¼-in. or 10.2-lb. material throughout. Top angle, 2 in. by 2 in. by ¼ in.

**Riveting.**—Tank to be single-riveted, ⅝-in. rivets, 2-in. pitch throughout, and to be provided with two (2) 2-in. flanges, located as close to the bottom as possible and 12 in. apart.

### One (1) Caustic Tank, 6 ft. Diameter by 5 ft. High, Open Top

**Material.**—To be constructed throughout of ¼-in. plate. Top angle, 2 in. by 2 in. by ¼ in.

**Riveting.**—Tank to be single-riveted throughout, ⅝-in. rivets, 2-in. pitch, and to be provided with two 2-in. flanges as close to bottom as possible, 12 in. apart.

### Two (2) Agitators, 25 ft. in Diameter by 35 ft. High

**Dimensions.**—25 ft. inside diameter by 35 ft. high in the shell.

**Material.**—Shell of agitator to be made up as follows:

First ring shall be of ½-in. or 20.4-lb. material.

Second ring, ½-in. or 20.4 lb. material.

Third ring, ⅜-in. or 15.3-lb.

Fourth ring, 5/16-in. or 12.75-lb.

Fifth and sixth rings, ¼-in. or 10.2-lb.

Shell to be made in five plates to each ring.

Around the bottom of the first ring, on both inside and outside, will be provided two 6-in. by 4-in. by ½-in. angles for riveting to the shell.

The cone bottom of agitator shall be made up of ten (10) or more sketch plates and one circular center plate of ½-in. or 20.4-lb. steel

Cone to drop about 30° or about 7 ft. 2 in. and be flanged for connection to shell. Cone is supported by ten (10) or more 4-in. wrought-iron pipes, to be furnished by the purchaser.

Riveted to each sketch plate of cone is a cast-iron bracket, to receive pipe supports, also cast-iron foundation plate for each pipe support. To the bottom of the cone is riveted one cast-iron nozzle, 8 in. in diameter, and to this is bolted one (1) 8-in. cast-iron flange, tapped for 6-in. pipe.

**Roof.**—Roof shall be of umbrella type, 5/16-in. or 7.65-lb., made up of sixteen plates, fifteen radial and one (1) center plate. The rise of the roof shall be about 30 in. Roof to be attached to the top ring of agitator by bolting to 2½-in. by 2½-in. by 5/16-in. angle, which is riveted around outside at the top. Roof will have six explosion hatches, 18 in. square, made up of 2-in. by 2-in. by ¼-in. angle with 3/16-in. plate cover. Each hatch will be provided with door, properly attached to prevent hatches from remaining open in case of an explosion.

**Opening.**—In the first ring of the agitator an opening will be provided for going in under the cone, 2 ft. 6 in. wide by 6 ft. 6 in. high, and will be reinforced on the inside by 2-in. by 2-in. by ¼-in. angle, no door being provided.

**Balcony.**—Around the agitator, approximately 3 ft. 2 in. down from the top, will be placed a balcony, 2 ft. 6 in. wide, made up as follows:

Floor shall be made up of plank, 1¾ in. thick by sketch, each plank bolted to balcony angle with one bolt at each end.

Balcony floor shall be supported by fifteen brackets made up of 2-in. by 2-in. by ¼-in. angle.

Connecting the agitator shell to floor of balcony, there is a 2-in. by 2-in. by ¼-in. angle. At outer edges of floor and attached to bracket is 2-in. by 2-in. by ¼-in. angle. Railing shall be made up of 2-in. by 2-in. by ¼-in. angle, fifteen posts being used. The bracket for the balcony of this agitator is so made that it also forms the posts for the railing. The agitators are to be placed 50-ft. centers and the necessary connecting steel platform with hand rail and steel plate floor to be provided. Also one steel stairway with a 1½ in. angle iron hand rail on each side is to be provided, running from the ground level to the center of the platform.

**Flanges.**—No flanges are to be furnished with agitator.

**Riveting.**—Bottom angle to first ring to be double-riveted, ⅞-in. rivets, 6-in. pitch.

First ring verticals, double-riveted, ⅞-in. rivets, 4-in. pitch.
Second ring to first ring, single-riveted, ⅞-in. rivets, 2½-in. pitch.
Second ring verticals, double-riveted, ⅞-in. rivets, 4-in. pitch.
Third ring to cone, single-riveted, ⅞-in. rivets, 2½-in. pitch.
Cone to second ring, double-riveted, ⅞-in. rivets, 3½-in. pitch.
Third ring verticals, double-riveted, ¾-in. rivets, 3⅛-in. pitch.
Fourth to third ring, single-riveted, ¾-in. rivets, 2¼-in. pitch.
Fourth ring verticals, double-riveted, ⅝-in. rivets, 2½-in. pitch.
Fifth to fourth ring, single-riveted, ⅝-in. rivets, 2-in. pitch.
Fifth ring verticals, double-riveted, ⅝-in. rivets, 2½-in. pitch.
Sixth ring to fifth ring, single-riveted, ⅝-in. rivets, 2-in. pitch.
Sixth ring verticals, double-riveted, ⅝-in. rivets, 2½-in. pitch.
Cone seams, double-riveted, ⅞-in. rivets, 3½-in. pitch.
Roof seams, single-riveted, 7/16-in. rivets, 1½-in. pitch.

Top angle to sixth ring, $\frac{5}{8}$-in. rivets, 4-in. pitch.

Rivets shall be provided of sufficient length for driving flat in the skirt of the agitator and flat for connection of cone to second ring; countersunk on the inside of cone and from cone up to the top.

**Shearing.**—All caulking edges shall be rotary bevel-sheared for inside caulking.

**Shipment.**—Agitator to be completely fitted up at works, match marked and shipped knocked down. Purchaser to erect.

## One (1) Condenser Box

**Dimensions.**—35 ft. long, 16 ft. 6 in. wide over all and 8 ft. deep; divided into two compartments, 8 ft. wide, 35 ft. long and 8 ft. deep.

**Shell.**—Shell of condenser shall be made up of $\frac{1}{4}$-in. or 10.2-lb. S.O.H. tank steel in three courses of three plates each, three plates being a part of the bottom and six plates forming the sides and a part of the bottom.

**Heads.**—Heads shall be made up of seven plates, each flanged for connection to bottom and end, and shall be of $\frac{1}{4}$-in. or 10.2-lb. S.O.H. tank steel.

**Partition.**—Partition shall be made up of seven plates, $\frac{1}{4}$-in. or 10.2-lb. S.O.H. tank steel, and shall be flanged for connection to bottom and end.

**Top Angle.**—Running completely around the condenser box at the top, and on the inside and across the partition, will be a 3-in. by 3-in. by $\frac{1}{4}$-in. angle riveted to shell, gusset plates being placed at the corners for stiffening purposes.

**Vertical Braces.**—On the outside, and running from the bottom of the condenser box 2 in. above the top, will be twenty-six 6-in., 8-lb. channel stiffeners, nine on each head and four on each end. Partition will also be stiffened with nine 6-in., 8-lb. channels.

**Cross-ties.**—Across the top of the condenser box and bolted through each channel will be $\frac{3}{8}$-in. by 2-in. cross-ties.

**Corner Angles.**—At each corner of each section of the condenser will be placed a $2\frac{1}{2}$-in. by $2\frac{1}{2}$-in. by $\frac{5}{16}$-in. angle stiffener.

**Flanges.**—On the bottom of each section of the condenser, 4 ft. from the end, will be located a 4-in. flange. In the center of the end of each section and as near the bottom as possible will be located a 3-in. flange. At the upper corner of each section in the end will be located a 3-in. flange.

**Riveting.**—All seams which are required to be water-tight will be punched for $\frac{7}{16}$-in. rivets, $1\frac{1}{2}$-in. pitch. Top angle will be punched for $\frac{7}{16}$-in. rivets, 4-in. pitch. Pitch of rivets in vertical stiffeners will vary from 3 in. to 6 in. in different places.

**Shearing.**—All plates to be rotary bevel-sheared for both inside and outside caulking.

**Shipment.**—After completion, condenser box shall be shipped in a completely knocked-down state, properly match marked for purchaser to erect.

## Seven (7) Condenser Boxes

**Dimensions.**—33 ft. 6 in. long, 25 ft. wide over all, 8 ft. deep, divided into four compartments, 8 ft. wide, 25 ft. long, 8 ft. deep.

**Shell.**—Shell of condenser shall be made up of ¼-in. or 10.2-lb. S.O.H. tank steel, in seven courses of three plates each, seven plates being a part of the bottom and fourteen plates forming the sides and part of the bottom.

**Heads.**—Heads shall be made up of five plates each, flanged for connection to the bottom and end, and shall be made of ¼-in. or 10.2-lb. S.O.H. tank steel.

**Partitions.**—Partitions shall be made up of five plates each, ¼-in. or 10.2-lb. S.O.H. tank steel, and shall be flanged for connection to bottom and ends.

**Top Angle.**—Running completely around the condensing box at the top, and on the inside and across each partition, will be a 3-in. by 3-in. by ¼-in. angle riveted to the shell, gusset plates being placed at the corners for stiffening purposes.

**Vertical Braces.**—On the outside, and running from the bottom of the condensing box to 2-in. above the top, will be thirty 6-in., 8-lb. channel stiffeners, eight on each end and seven on each head. Each partition will also be stiffened with seven 6-in., 8-lb. channels.

**Cross-ties.**—Across the top of the condensing box and bolted to each channel will be ⅜-in. by 2-in. cross-ties.

**Corner Angles.**—At each corner of each section of the condenser will be placed 2½-in. by 2½-in. by 5/16-in. angle stiffeners.

**Flanges.**—On the bottom of each section of the condenser, 4 ft. from the end, will be located a 4-in. flange. In the center of the end of each section and as near the bottom as possible will be located a 3-in. flange. At the upper corner of each section in the end will be located a 3-in. flange.

**Riveting.**—All seams which are required to be water-tight will be punched for 7/16-in. rivets, 1½-in. pitch. Top angle will be punched for 7/16-in. rivets, 4-in. pitch. Pitch of rivets in vertical stiffeners will vary from 3 in. to 6 in. at different places.

**Shearing.**—All plates to be rotary bevel-sheared for both inside and outside caulking.

**Shipment.**—After completion, condensing box shall be shipped in a completely knocked-down state, properly match marked for purchaser to erect.

### Six (6) Condenser Boxes

**Dimensions.**—25 ft. long, 8 ft. wide, 4 ft. deep, inside dimensions.

**Shell.**—Shell of condenser shall be of No. 6 or 8.29-lb. S.O.H. tank steel and shall be made up of five courses of one plate each, each plate forming the side and bottom.

**Heads.**—Each head of the condenser will be formed of one plate No. 6 or 8.29-lb. S.O.H. tank steel, flanged for connection to shell.

**Top Angle.**—Running completely around the condenser on the top and on the outside will be a 3-in. by 3-in. by ¼-in. angle, riveted to the shell, gusset plates being placed at the corners for stiffening purposes.

**Cross-ties.**—Across the top and equally spaced will be two cross-ties, ½-in. by 2½-in., bolted to top angle at each side with ⅝-in. bolts.

**Flanges.**—On the bottom, 4 ft. from the end, as near the side as possible, will be located one 4-in. flange. On the end, as near the bottom as possible, will be one 3-in. flange.

**Riveting.**—All seams which it is necessary to have water-tight will be punched for 7/16-in. rivets, 1½-in. pitch. Top angle will be punched for 7/16-in. rivets, 4-in. pitch.

**Shearing.**—All plates to be rotary bevel-sheared for inside and outside caulking.

**Shipment.**—After completion, condenser shall be shipped in a completely knocked-down state, properly match marked for purchaser to erect.

### Seven (7) Condenser Supports

Seven condenser supports for 33½-ft. by 25-ft. by 8-ft. condensers (see Fig. 263).

### One (1) Condenser Support

One condenser support for 35-ft. by 16½-ft. by 8-ft. condenser.

### Two (2) Crude Oil Stills

**Dimensions.**—12 ft. inside diameter of outside top course by 35 ft. long in the shell.

**Shell.**—Shell of still shall be made up as follows:

*Bottom.*—Bottom shall be made up of two plates, ½ in., or 20.4 lb., by 109 in. wide, still bottom steel. Plate toward front to be 25 ft. long; plate toward the back 10 ft. long.

*Roundabouts.*—Remainder of the shell forming the sides and top of still shall be made up of five plates, running girth-ways, of ⅜-in. or 15.3-lb. S.O.H. tank steel.

*Heads.*—Heads of stills shall be made up of two plates each, 12-in. or 20.4-lb. flange steel, and shall be dished to 15-ft. 6-in. radius and flanged for connection to shell.

**Dome.**—There shall be one dome, 36 in. diameter by 36 in. high, located in center of the center top course. Dome shall be made up of ⅜-in. or 15.3-lb. flange steel and shall be flanged for connection to shell. The head for dome shall be of ⅜-in. or 15.3-lb. flange steel and be

dished and flanged for connection to dome shell. To the dome head will be riveted one 10-in. steel flange.

**Neck Manheads.**—To the front and back heads of still will be attached one 20-in. neck manhead made up of ⅜-in. or 15.3-lb. flange steel. Manheads will be equipped with cover properly faced and made of 9/16-in. plate and fastened to the collar (which is riveted to the neck and also faced) with twenty-eight ¾-in. square neck bolts. Manheads will also be provided with crane for carrying cover.

**Yoke Manhead.**—Each still will have one 18-in. yoke manhead, located 36-in. from back end. Ring and cover of manhead shall be of cast iron. Yoke eyes and handles shall be steel forgings.

**Lugs.**—Each still shall have sixteen cast-iron lugs, eight on each side. Each lug shall be riveted to shell with ten ⅞-in. rivets.

**Flanges.**—Each still will be provided with the following flanges:

One 5-in., located on top. One 2-in., located on top. One 1-in., on side of dome toward front end. One 10-in., on top of dome. One 3-in., for tar plug. Two 1½-in., on rear head. One 1¼-in., on rear head.

**Tar Plug.**—Each still will be provided with a 3-in. tar plug with rod, brackets, handles and levers for operating same from back end of still; suitable stuffing box and gland shall also be provided on top of still for lifting rod to pass through.

**Riveting.**—All rivets shall be ¾-in. diameter. All longitudinal seams shall be double-riveted, 2.76-in. pitch. Cross seams of head shall be double-riveted, 2.76-in. pitch. Head, double-riveted to shell, 2¾-in. pitch. Girth seams, double-riveted, 2¾-in. pitch. Dome to shell, double-riveted, 2¾-in. pitch. Remainder of dome, single-riveted, ¾-in. rivets, 2¼-in. pitch. All rivets will be provided of sufficient length for driving full head on the outside of still with the following exceptions: Lugs, the rivets for which will be provided of sufficient length for driving flat on the inside; also neck manhead, yoke manhead and stuffing box.

**Shearing.**—All seams shall be rotary bevel-sheared for outside caulking. Punched out holes for yoke manhead, neck manhead, etc., shall be neatly bevel-chipped for caulking.

**Shipment.**—After completion, stills shall be shipped in a completely knocked-down condition, properly match marked for purchaser to erect.

## Twenty (20) Crude Oil Stills

**Dimensions.**—8 ft. inside diameter of outside top course by 20 ft. long in the shell.

**Shell.**—Shell of still shall be made up as follows:

*Bottom.*—Bottom shall be made up of one plate, ½-in. or 20.4-lb. by 99-in. wide, still bottom steel.

*Roundabouts.*—Remainder of the shell forming the sides and top of still shall be made up of three plates, running girth-ways, of ⅜-in., or 15.3-lb., S.O.H. tank steel.

*Heads.*—Heads of stills shall be made up of one plate, each ½-in., or 20.4-lb., flange steel, and shall be dished to 9-ft. 2-in. radius and flanged for connection to shell.

**Dome.**—There shall be one dome, 24 in. diameter by 20 in. high, located in center of the center top course. Dome shall be made up of ⅜-in., or 15.3-lb., flange steel, and shall be flanged for connection to shell. The head for dome shall be of ⅜-in., or 15.3-lb., flange steel and be dished and flanged for connection to dome shell. To the dome head will be riveted one 8-in. steel flange.

**Neck Manheads.**—To the front head of still will be attached one 20-in. neck manhead, made up of ⅜-in., or 15.3-lb., flange steel. Manhead will be equipped with cover, properly faced and made of 9/16-in. plate, and fastened to the collar (which is riveted to the neck and also faced) with twenty-eight ¾-in. square neck bolts. Manhead will also be provided with crane for carrying cover.

**Yoke Manhead.**—Each still will have one 18-in. yoke manhead, located 35 in. from back end. Ring and cover of manhead shall be of cast iron. Yoke eyes and handles shall be steel forgings.

**Lugs.**—Each still shall have twelve cast-iron lugs, six on each side. Each lug shall be riveted to shell with ten ⅞-in. rivets.

**Flanges.**—Each still shall be provided with the following flanges:

One 5-in., located on top. One 2-in., located on top. One 1-in., on side of dome toward front end. One 8-in., on top of dome. One 3-in., for tar plug. Two 1½-in., on rear head. Two 5-in. C.S. vapor flanged, on rear head. One 1-in. flange, on front head, 1 ft. from bottom and 1 ft. from M.H.

**Tar Plug.**—Each still will be provided with a 3-in. tar plug with rod, brackets, handles and levers for operating same from back end of still; suitable stuffing box and gland shall also be provided on top of still for lifting rod to pass through.

**Riveting.**—All rivets shall be ¾ in. diameter. All longitudinal seams shall be double-riveted, 2.76-in. pitch. Head, double-riveted to shell, 2¾-in. pitch. Girth seams, double-riveted, 2¾-in. pitch. Dome to shell, double-riveted, 2¾-in. pitch. Remainder of dome, single-riveted, ¾-in. rivets, 2¼-in. pitch. All rivets will be provided of sufficient length for driving full head on the outside of still, with the following exceptions: Lugs, the rivets for which will be provided of sufficient length for driving flat on the inside; also neck manhead, yoke manhead and stuffing box.

**Shearing.**—All seams shall be rotary bevel-sheared for outside caulking. Punched out holes for yoke manhead, neck manhead, etc., shall be neatly bevel-sheared for caulking.

**Shipment.**—Stills shall be shipped built up complete, with possible exception of the drum, which the railroad company may require to be left off.

## Eight (8) Steam Stills

**Dimensions.**—Stills shall be 8 ft. inside diameter by 8 ft. long in the shell, with dished heads (see Fig 242).

**Material.**—Heads to be made up of 5/16-in., or 12.75-lb., plate, flanged and dished for riveting to shell of still. Shell of still shall be made up of one ring, 1/4-in., or 10.2-lb., plate.

**Manheads.**—Each still will have one 18-in. yoke manhead located on top, 2 ft. back from one end. Ring and cover of manhead shall be of cast iron. Yoke eyes and handles shall be of steel forgings.

**Flanges.**—The following flanges are furnished with still:

One 2-in., located on shell on bottom.

Two 2-in., located on shell on top.

One 8-in., located on top of shell above the center.

Two 4-in., located on one head.

One still to have two 1-in. flanges, located 16 in. from the top and bottom of head with the 4-in. flanges.

All stills to have 1-in. flange on same head as 4-in.

**Riveting.**—The heads of stills to be single-riveted with 5/8-in. rivets, 2-in. pitch. Longitudinals to be double-riveted with 5/8-in. rivets, 2½-in. pitch. Rivets will be driven full heads.

**Shearing.**—All plates to be rotary bevel-sheared for inside and outside caulking.

**Shipment.**—After completion, stills will be shipped completely built up.

**Painting.**—Stills to be given one coat of paint on the outside before shipment.

## Eight (8) Steam Still Drums

Eight steam still drums, 30 in. diameter by 7 ft. high, to have necessary 8-in. and 1½-in. flanges and baffle plates as called for (see Fig. 249).

## Three (3) Blow Tanks

**Dimensions.**—Blow tanks shall be 36-in. inside diameter by 60-in. long in the shell.

**Shell.**—Shell of blow tanks to be made up of one course 1/4-in., or 10.2-lb., S.O.H. tank steel.

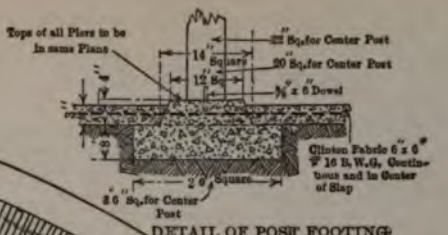
**Heads.**—Heads shall be made up of one plate, each 5/16-in., or 12.75-lb., S.O.H. tank steel, and shall be dished and flanged for connection to shell. One head will be backed in, thus not making a manhead necessary.

**Flanges.**—One 2-in. flange shall be located on the top near one end. One 2-in. flange shall be located on the bottom near the opposite end.

**Riveting.**—Longitudinal seams shall be double-riveted, 5/8-in. rivets, 2½-in. pitch. Heads to shell shall be single-riveted, 5/8-in. rivets, 2-in. pitch.

Height Schedu

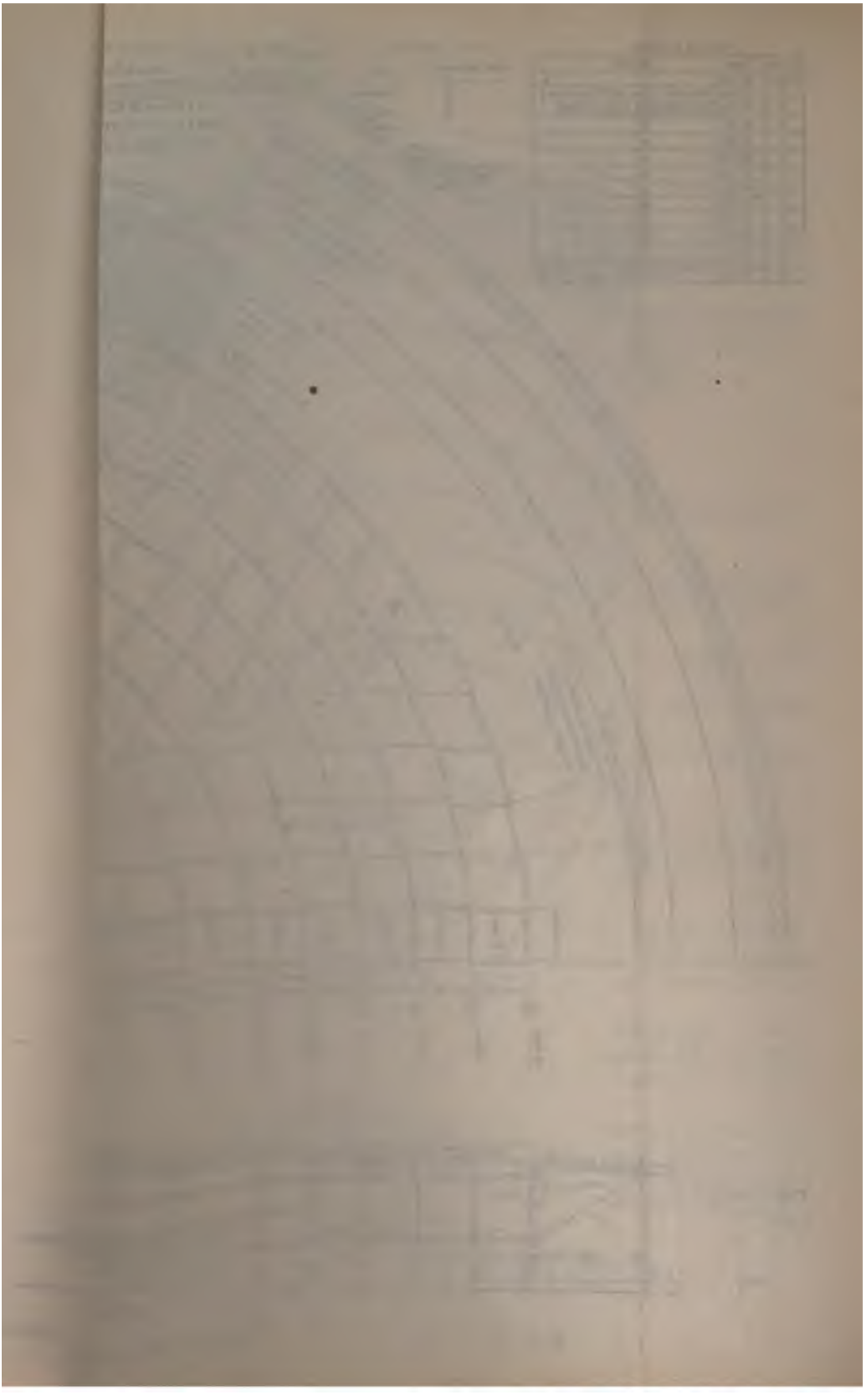
| Row No. | Height | Re |
|---|---|---|
| Center | 33' 0¾" | |
| 1 | 33' 0¾" | Posts Cut 4 Lo |
| 2 | 32' 11¾" | Post Cut to sam |
| 3 | 32' 10" | do |
| 4 | 32' 8⅜" | do |
| 5 | 32' 7⅛" | do |
| 6 | 32' 6¾" | do |
| 7 | 32' 5" | do |
| 8 | 32' 3⅜" | do |
| 9 | 32' 2⅛" | do |
| 10 | 32' 1¼" | do |
| 11 | 32' 0" | do |
| 12 | 32' 10⅜" | Except One In E |
| 13 | 24' 8⅛" | " Two on |




DETAIL OF POST FOOTING




(Facing page 696).



**Shearing.**—Plates shall be rotary bevel-sheared for outside caulking.

**Caulking.**—Blow tanks shall be well caulked with a round nose caulking tool.

**Testing.**—Blow tanks shall be tested and made water-tight at 100 lb. pressure.

**Shipment.**—After completion, blow tanks shall be shipped built up, all complete.

## RESERVOIRS

Concrete-lined earthen reservoirs are used for the storage of petroleum in the Mid-Continent, Gulf and California fields. Fig. 212 gives the details of construction of a typical reservoir in the Kern River field, Cal. The specifications of this 750,000-bbl. reservoir were as follows:

| | |
|---|---|
| Diameter at bottom | 462 ft. |
| Diameter at top | 528 ft. |
| Depth | 22 ft. |
| Inside slope | 1½ horizontal, 1 vertical |
| Outside slope | 1½ horizontal, 1 vertical |
| Top of embankment | 11 ft. |
| Slope of top | 6 in. in 11 ft. |
| Area of bottom | 167,600 sq. ft. |
| Area of inside slope | 61,600 sq. ft. |
| Total area inside | 229,200 sq. ft. |
| Capacity of tank | 750,000 bbl. (42 gal.) |

The costs were:

| | |
|---|---|
| Total cost, per barrel capacity, to build reservoir. | $0.10 to $0.11 |
| Labor: | |
| Earthwork (per yard) | $0.30 to $0.50 |
| Concrete (per yard) | 0.70 to 0.80 |
| Roofing per M | 5.00 to 6.00 |

## TANKS

Steel tankage for oil storage is usually of the following dimensions:

| Capacity | Diameter | Height |
|---|---|---|
| 55,000 barrels | 114 ft. 6 in. | 30 ft. |
| 37,000 barrels | 94 ft. 0 in. | 30 ft. |
| 30,000 barrels | 86 ft. 0 in. | 30 ft. |
| 20,000 barrels | 70 ft. 0 in. | 30 ft. |
| 20,000 barrels | 77 ft. 0 in. | 25 ft. |
| 10,000 barrels | 49 ft. 7 in. | 30 ft. |
| 10,000 barrels | 54 ft. 0 in. | 25 ft. |
| 5,000 barrels | 43 ft. 0 in. | 20 ft. |

There is considerable variation in specifications and cost. The following specifications represent probably the heaviest and most substantial construction of a 55,000-bbl. tank with steel roof.

*Dimensions.*—One hundred and fourteen feet six inches (114 ft. 6 in.) diameter by thirty feet (30 ft.) high

*Thickness of Material and Riveting.—*

| Description of part | | Diameter of rivets | | Rows of rivets | |
|---|---|---|---|---|---|
| Ring | Dimensions, pounds per square foot | Vertical, inches | Horizontal, inches | Vertical | Horizontal |
| First | 23 | 7/8 | 3/4 | Triple | Single |
| Second | 19 | 3/4 | 3/4 | Triple | Single |
| Third | 14 | 3/4 | 3/4 | Double | Single |
| Fourth | 12 | 5/8 | 5/8 | Double | Single |
| Fifth | 10 | 3/8 | 3/8 | Single | Single |
| Sixth | 8 | 3/8 | 3/8 | Single | Single |

Bottom sketch plates, No. 4 gauge, 3/8-in. rivets, single.
Bottom rectangular plates, No. 6 gauge, 3/8-in. rivets, single.
Bottom angles, 4-in. by 4-in. by 1/2-in., 3/4-in. rivets, single.
Top angles, 2 1/2-in. by 2 1/2-in. by 5/16-in., 7/16-in. rivets, single.

| Description of part | Steel, thickness | Rivets, vertical seams | Rivets, horizontal seams |
|---|---|---|---|
| First ring | 9/16 in. | 7/8 in. triple | 3/4 in. single |
| Second | 15/32 in. | 3/4 in. triple | 3/4 in. single |
| Third | 11/32 in. | 3/4 in. double | 3/4 in. single |
| Fourth | 5/16 in. | 5/8 in. double | 5/8 in. single |
| Fifth | 1/4 in. | 7/16 in. double | 7/16 in. single |
| Sixth | 7/32 in. | 3/8 in. single | 3/8 in. single |
| Bottom | 1/4 in. | ..... | 1/2 in. single |

Top angles, 3 1/2-in. by 3 1/2-in. by 3/8-in., 7/16-in. rivets, single.
Bottom angles, 4-in. by 4-in. by 1/2-in., 3/4-in. rivets, single.
Roof, 1/8-in. thick, 5/16-in. rivets, single.

The cost of such tanks, including grading and piping, is shown by a contract at Coalinga, Cal., in 1913 to be $0.346 per barrel (20 tanks, $380,000). Lighter tanks with wooden roofs[1] are more

[1] Steel roofs are sometimes installed on refinery tanks, but wooden roofs are frequently used both in the refinery and in the field.

common and cost 23 cts. to 25 cts. per barrel, plus the foundation cost. Solid earth foundation is necessary to prevent seams from opening.

The Hammond Iron Works, of Warren, Pa., has courteously supplied the following dimensions and specifications for tankage.

TABLE LV.—DIMENSIONS AND SPECIFICATIONS FOR TANKAGE

| (42-gal. Bbl.) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Nominal capacity in barrels | 25 | 50 | 75 | 100 | 150 | 200 | 250 | 300 | 350 | 400 |
| Actual capacity in barrels | 25 | 53 | 79 | 102 | 168 | 201 | 274 | 314 | 358 | 404 |
| Diameter of tank in feet | 6 | 8 | 9 | 9 | 10 | 12 | 14 | 15 | 16 | 17 |
| Height of tank in feet | 5 | 6 | 7 | 9 | 12 | 10 | 10 | 10 | 10 | 10 |
| Number of rings in shell | 1 | 1 | 2 | 2 | 3 | 2 | 2 | 2 | 2 | 2 |
| Thickness of first ring B.W.G. | 8 | 8 | 8 | 7 | 7 | 7 | 7 | 7 | 7 | 7 |
| Thickness of second ring B.W.G. | ..... | ..... | 8 | 8 | 8 | 8 | 8 | 8 | 8 | 8 |
| Thickness of third ring B.W.G. | ..... | ..... | ..... | ..... | 9 | | | | | |
| Thickness of bottom ring B.W.G. | 8 | 8 | 8 | 8 | 8 | 8 | 8 | 8 | 8 | 7 |
| Size of bottom angle iron | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2½×5/16 |
| Size of top angle iron | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ |
| Thickness of sheets for light nailed roof | ..... | ..... | ..... | 24 | 24 | 24 | 24 | 24 | 24 | 24 |
| Thickness of sheets for tight roof | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

| (42-gal. Bbl.) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Nominal capacity in bbl. | 500 | 1,000 | 2,000 | 2,500 | 3,000 | 4,000 | 5,000 | 10,000 |
| Actual capacity in barrels | 559 | 1,119 | 2,186 | 2,518 | 3,147 | 4,043 | 5,173 | 10,198 |
| Diameter of tank in feet | 20 | 20 | 25 | 30 | 30 | 34 | 43 | 54 |
| Height of tank in feet | 10 | 20 | 25 | 20 | 25 | 25 | 20 | 25 |
| Number of rings in shell | 2 | 4 | 5 | 4 | 5 | 5 | 4 | 5 |
| Thickness of first ring B.W.G. | 7 | 6 | 5 | 5 | 5 | 4 | 3 | 3 |
| Thickness of second ring B.W.G. | 8 | 7 | 6 | 6 | 6 | 5 | 4 | 3 |
| Thickness of third ring B.W.G. | ..... | 8 | 7 | 7 | 7 | 6 | 5 | 4 |
| Thickness of fourth ring B.W.G. | ..... | 8 | 8 | 8 | 8 | 7 | 6 | 5 |
| Thickness of fifth ring B.W.G. | ..... | ..... | 8 | ..... | 8 | 7 | ..... | 6 |
| Thickness of bottom | 7 | 7 | 7 | 7 | 7 | 7 | 6 | 6 |
| Size of bottom angle iron | 2½×5/16 | 2½×5/16 | 2½×5/16 | 2½×5/16 | 2½×5/16 | 2½×⅜ | 2½×⅜ | 2½×⅜ |
| Size of top angle iron | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×¼ |
| Thickness of sheets for light nailed roof | 24 | 24 | 24 | 24 | 24 | 24 | 24 | 24 |
| Thickness of sheets for tight roof | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

| (42-gal. Bbl.) | | | | | | | |
|---|---|---|---|---|---|---|---|
| Nominal capacity in barrels | 15,000 | 20,000 | 25,000 | 30,000 | 35,000 | 40,000 | 50,000 |
| Actual capacity in barrels | 15,235 | 20,199 | 25,531 | 31,037 | 35,520 | 40,304 | 50,778 |
| Diameter of tank in feet | 66 | 76 | 78 | 86 | 92 | 98 | 110 |
| Height of tank in feet | 25 | 25 | 30 | 30 | 30 | 30 | 30 |
| Number of rings in shell | 5 | 5 | 6 | 6 | 6 | 6 | 6 |
| Thickness of first ring B.W.G | 3 | 2 | 2 | 0 | 00 | 00 | 000 |
| Thickness of second ring B.W.G | 3 | 3 | 3 | 1 | 0 | 00 | 00 |
| Thickness of third ring B.W.G | 4 | 4 | 4 | 2 | 1 | 0 | 0 |
| Thickness of fourth ring B.W.G | 5 | 5 | 5 | 3 | 2 | 1 | 1 |
| Thickness of fifth ring B.W.G | 6 | 6 | 6 | 4 | 3 | 2 | 2 |
| Thickness of sixth ring B.W.G | ..... | ..... | 7 | 5 | 4 | 3 | 3 |
| Thickness of bottom B.W.G | 6 | 6 | 6 | 6 | 6 | 5 | 5 |
| Size of bottom angle iron | 3×5/16 | 3×5/16 | 3×5/16 | 4×½ | 4×½ | 4½×½ | 4½×½ |
| Size of top angle iron | 2×¼ | 2×¼ | 2×¼ | 2×¼ | 2×5/16 | 2×5/16 | 2×5/16 |
| Thickness of sheets for light nailed roof | 24 | 24 | 24 | 24 | 24 | 24 | 24 |
| Thickness of sheets for tight roof | 12 | 12 | 10 | 10 | 10 | 10 | 10 |

The following is a copy of the standard specifications in general use by oil tank builders.

SPECIFICATIONS

Accompanying and forming part of proposal for two steel storage tanks.

**Dimensions.**—95 ft. in diameter by 30 ft. high.

**Capacity.**—37,878 bbl. of 42 gal. each.

**Plates.**—Plates in general to be best grade of class "A" steel. Plates requiring forging or flanging to be soft open-hearth or flange steel quality.

**Structural Shapes.**—Angles and structural shapes to be manufacturers' standard specifications of medium steel.

**Castings.**—All cast-iron parts to be good quality gray iron. Steel castings to be of good quality open-hearth or basic steel and free from injurious defects.

**Construction.**—Bottom rectangular plates, 8.29 lb. per square foot, single-riveted, 7/16-in. rivets, 1½-in. pitch.

Bottom sketch plates, 9.56 lb. per square foot, single-riveted, 7/16-in rivets, 1½-in. pitch.

Bottom angle, 4 in. by 4 in. by ½ in., single-riveted, ¾-in. rivets, 2.4-in. pitch.

First ring plates, 17.85 lb. per square foot. Vertical seams, double-riveted, ¾-in. rivets, 2 1/16-in. pitch.

Second ring plates, 15.3 lb. per square foot. Vertical seams, double-riveted, ¾-in. rivets, 2⅞-in. pitch.

Third ring plates, 11.47 lb. per square foot. Vertical seams, double-riveted, ⅝-in. rivets, 2½-in. pitch.

Fourth ring plates, 10.2 lb. per square foot. Vertical seams, double-riveted, 7/16-in. rivets, 1½-in. pitch.

Fifth ring plates, 8.92 lb. per square foot. Vertical seams, double-riveted, 7/16-in. rivets, 1⅝-in. pitch.

Sixth ring plates, 8.29 lb. per square foot. Vertical seams, double-riveted, 7/16-in. rivets, 1⅝-in. pitch.

Shell to be constructed of 19 plates to each ring. All horizontal seams, single-riveted.

Top angle to be 2½ in. by 2½ in. by 5/16 in., single-riveted, 7/16-in. rivets, 1½-in. pitch.

**Manhole.**—One steel manhole, 20 in. in diameter, in center of first ring.

**Flanges.**—The following flanges, or their equal in monetary value, to be furnished: two 6-in., one 4-in., two 2-in.

Location of above flanges to be furnished by the customer within ten days from date of signing contract.

**Ladder.**—No ladder to be furnished.

**Swing-pipe.**—Tank to be furnished with a 6 in. diameter swing-pipe, our standard, complete with wire rope and windlass, including gate valve. Windlass to be located on roof of tank near the side and to be gas-tight. Valve to be Ludlow IBBM valve.

**Stairs.**—Standard steel stairs to be furnished.

**Paint.**—Paint to be good quality red metallic.

**Punching.**—All holes to be accurately laid out and punched true, using punches 1/16 in. larger in diameter than the rivets.

**Shearing.**—All edges that require caulking are to be carefully beveled.

**Riveting.**—Rivets, where possible, are to be driven cold. Rivets ⅝-in. and larger to be driven hot. All shell rivets to be driven flat, heads on the outside. All bottom rivets to be driven flat, heads on inside of bottom.

Spacing of rivets to be such as will guarantee the tank when completed to withstand safely all internal pressure when filled with oil.

**Caulking.**—All seams to be well caulked, metal to metal.

**Testing.**—Purchaser to furnish free of cost, promptly when desired by seller, water in sufficient quantity to insure quick filling, piped into each piece of equipment for testing, or any other purpose. Any delays in furnishing water for test, the purchaser to pay field foreman's wages during such delay.

When test has been made, purchaser shall accept each piece of equipment in writing, such acceptance to be final.

In the event purchaser fails to furnish water for testing as stated above and the equipment has been completed, it will then be understood that same will be accepted without test.

**Painting.**—After completion, the outside of shell is to be given two coats of red metallic paint.

**Foundations.**—Solid and level foundations no less than 10 ft. greater than size of equipment to be furnished by the purchaser and there shall be at least 5 ft. clearance outside of any part of the equipment. Founda-

tions not to be over 2 ft. above the level of the surrounding ground, same to be ready when needed by the seller. Purchaser to notify seller in writing ten days prior to completion of foundation. If foundations are not ready on arrival of field foreman, purchaser to pay field foreman's wages for such time as he is unable to work on same.

**Site.**—The site for each piece of equipment shall be practically on a level with and not over 1,000 ft. from a railroad siding and guaranteed by the purchaser free of inundation until such equipment has been accepted. The ground over which this material must be conveyed from the railroad siding to be generally level, solid and free of obstructions.

**Steel Plate Roof.**—Roof to be cone type with a rise of about 6 ft.

**Plates.**—To be best grade of class "A" steel and to be No. 10 gauge thick, or 5.5 lb. per square foot.

**Riveting.**—All seams to be single-riveted. Rivets to be ⅜-in. diameter and spaced approximately 1½-in., center to center.

**Caulking.**—Roof to be caulked and made gas-tight.

**Supports.**—Roof to be supported by twelve 9-in., 21-lb., I-beam posts, connected at the tops by 8-in., 11¼-lb. bent channels which support the 5-in., 9¾-lb., I-beam rafters. Rafters in two rows with 57 in outer row and 24 in inner row.

**Hatch.**—Steel hatch, size 20 in. in diameter, located near the side, also on 8-in. flange in center of roof.

**Painting.**—Roof, when completed, to receive two coats of paint on the outside, same quality as used on shell.

**Delivery.**—We will commence shipment in about ——— days from the acceptance of our proposition and the settling of all details pertaining to same. We will start erection as soon as possible after arrival of material at destination. Delivery and erection subject, however, to strikes, fires, accidents or other causes beyond our control.

**Erection.**—This equipment to be erected by ———— Company on premises of the purchaser at ————.

**Acceptance.**—The proposition of which these specifications are a part, is for prompt acceptance and subject to change without notice.

**Price and Terms.**—Prices and terms as per accompanying letter dated ————, based on present prices of material and labor. All understandings outside of these specifications must be agreed to by both parties thereto in writing.

Figure 213 shows the details of a barreling tank, 36 ft. in diameter and 10 ft. high. Fig. 215 shows six tanks of this design, built by the Treadwell Construction Company, of Midland, Pa.

Figures 216 and 217 give the specifications for 35,000- and 55,000-bbl. tanks constructed recently by the Riter-Conley Manufacturing Company of Pittsburgh, Pa. A 55,000-bbl.

capacity storage tank, built for the United States Navy by the Treadwell Company, is shown in Fig. 218.

A 55,000-bbl. storage tank for crude oil, provided with an ordinary wooden roof with sheet-steel covering, is illustrated in

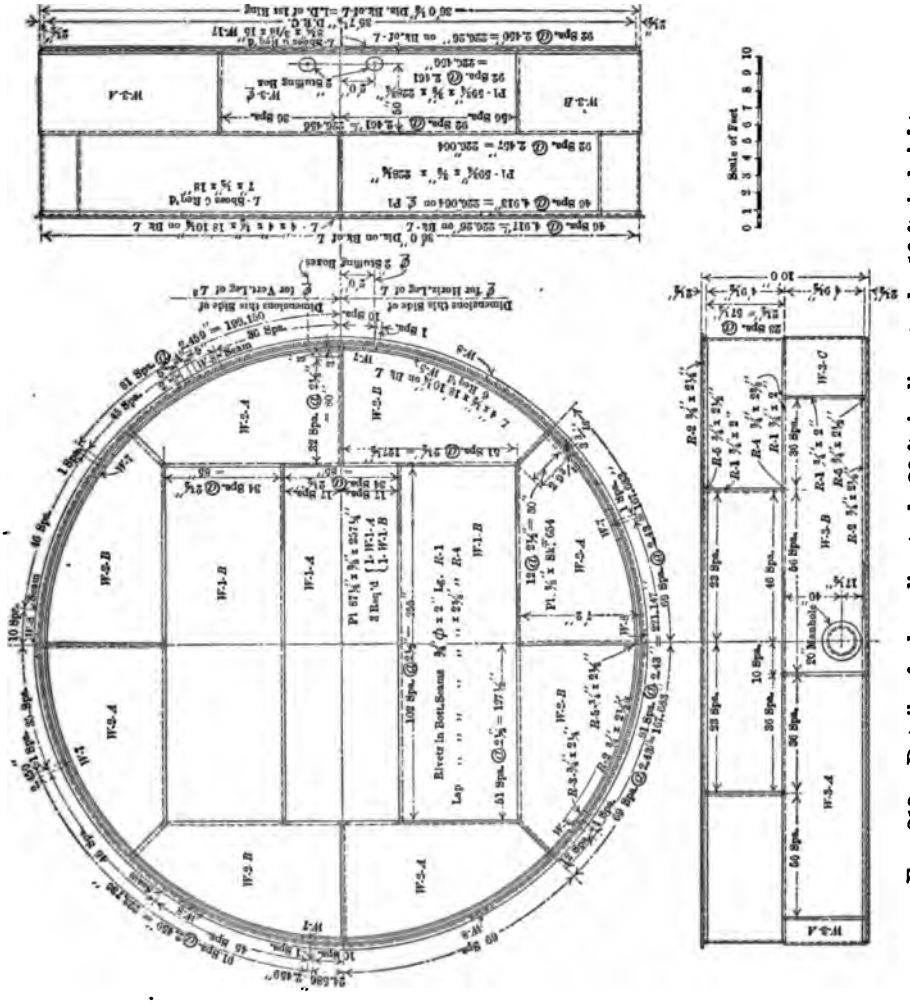


FIG. 213.—Details of a barreling tank, 36 ft. in diameter by 10 ft. in height.

Fig. 219. When an all steel roof is preferred, this is supported with a structural steel framework, as shown in Fig. 220; this frame is then covered with sheets, ⅛ in. thick, riveted and caulked. These illustrations are published through the courtesy

of the Hammond Iron Works, of Warren, Pa. In some localities certain refineries use storage tanks with water-top roofs; this is

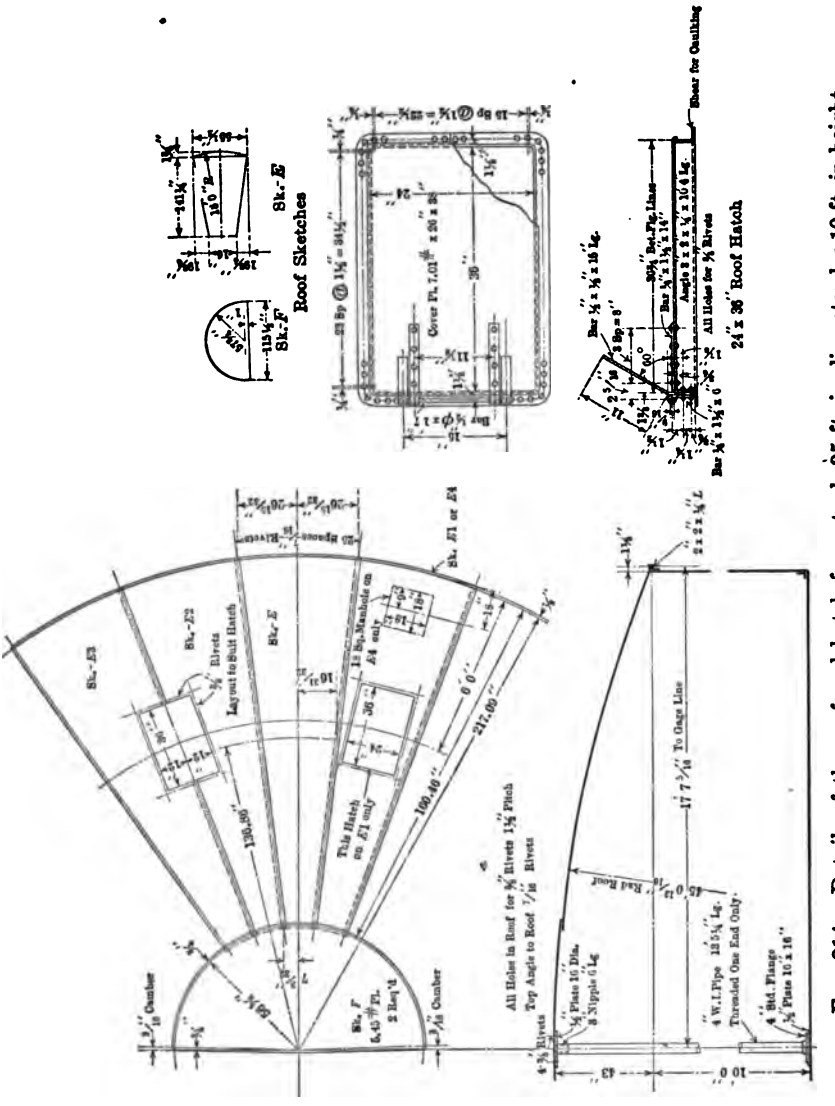



FIG. 214.—Details of the roof and hatch for a tank 35 ft. in diameter by 10 ft. in height.

said to be a very satisfactory method for the storage of light products. Fig. 221 shows this type of tank.

A horizontal tank, 6 ft. in diameter and 24 ft. in length, is detailed in Fig. 222.

Figures 223 and 224 give the details of an acid storage tank, 8 ft. in diameter and 20 ft. in height.

Figures 225 and 226 show the construction of asphalt-plant tanks.

Fig. 227 exhibits two receiving tanks, 9 ft. in diameter by 48 ft. in length.

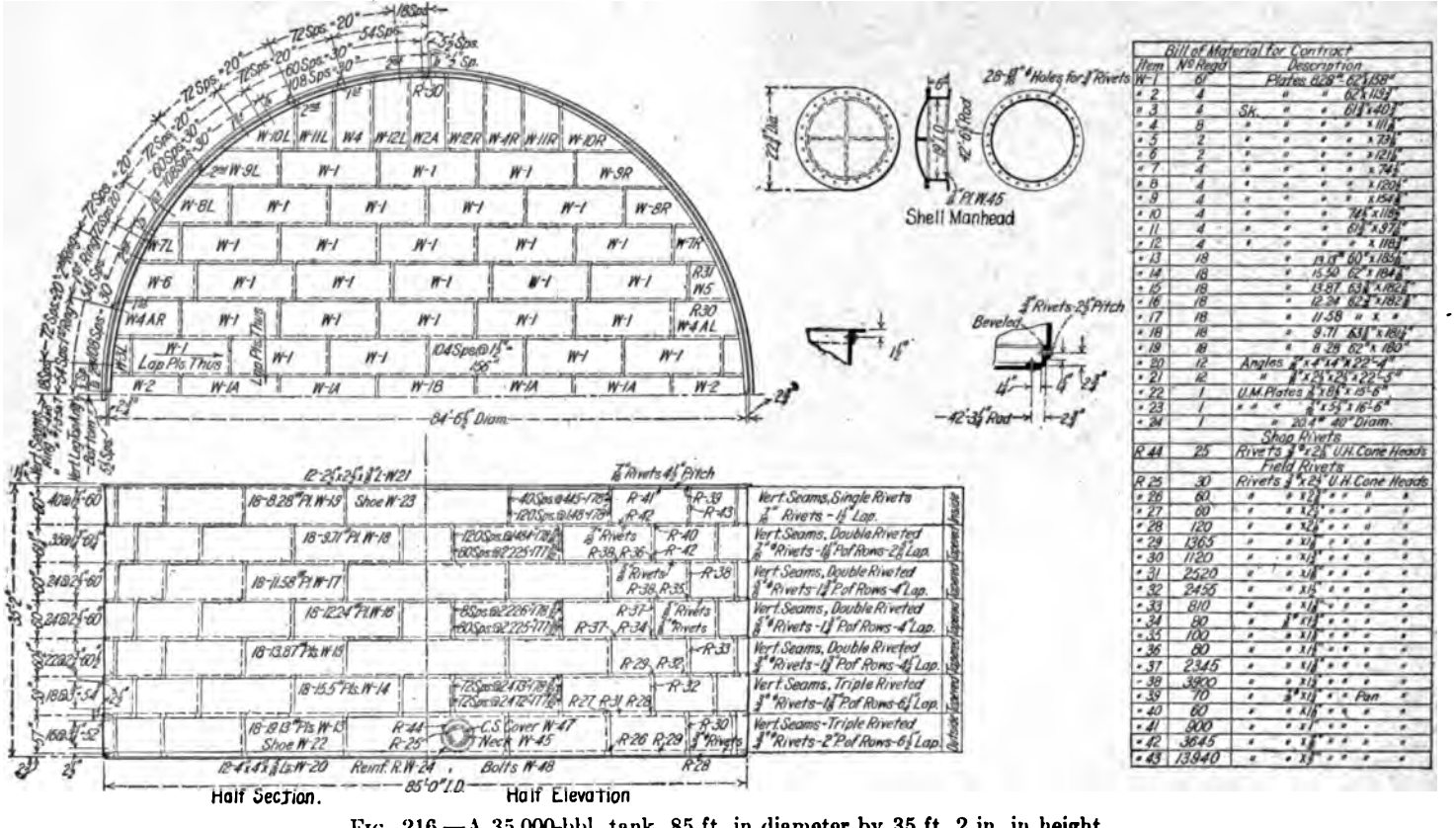
**Measurement.**[1]—The following formulas are in use for computing the capacities of circular tanks:




FIG. 215.—Six barreling tanks, 36 ft. in diameter by 10 ft. in height, constructed for the Huasteca Petroleum Company.

D = diameter.
C = circumference.
U. S. G. = United States gallons of 231 cu. in.
Imp. G. = Imperial gallons of 277.274 cu. in.
D in inches squared × 0.0034 = U. S. G. per inch.
D in inches squared × 0.00283257 = Imp. G. per inch.
D in feet squared × 0.011656 = 42 U. S. G. bbl. per inch.
C in feet squared ÷ 20.1586 = U. S. G. per inch.
C in feet squared × 0.04960677 = U. S. G. per inch.
C in feet squared × 0.00118111 = 42 U. S. G. bbl. per inch.
C in feet squared × 0.00099213 = 50 U. S. G. bbl. per inch.
C in feet squared × 0.041327896 = Imp. G. per inch.
Imp. G. × 1.20032 = U. S. G.
U. S. G. × 0.83311 = Imp. G.

[1] On computing the capacities of circular tanks, vertical and horizontal, see D. O. TOWL's "Tanks," **1901**, pp. 3 and 18. On gauging storage tanks, see OGDEN, *J. Ind. Eng. Chem.*, **8** (1916), 58; and HOWELL, *ibid.*, 286.




| Bill of Material for Contract | | |
|---|---|---|
| Item | No. Regd | Description |
| W-1 | 61 | Plates 0.25" = 62.1358# |
| " 2 | 4 | " " 62" x 113" |
| " 3 | 4 | Sk. " " 61⅞" x 140" |
| " 4 | 8 | " " " " x 111⅛" |
| " 5 | 2 | " " " " x 73" |
| " 6 | 2 | " " " " x 121" |
| " 7 | 4 | " " " " x 74" |
| " 8 | 4 | " " " " x 120" |
| " 9 | 4 | " " " " x 154" |
| " 10 | 4 | " " " " 74" x 119" |
| " 11 | 4 | " " " " 69" x 97" |
| " 12 | 4 | " " " " x 118" |
| " 13 | 18 | " 13.15 " 60" x 185" |
| " 14 | 18 | " 15.20 " 62" x 184" |
| " 15 | 18 | " 13.87 " 63" x 182" |
| " 16 | 18 | " 12.24 " 82" x 182" |
| " 17 | 18 | " 11.58 " " x " |
| " 18 | 18 | " 9.71 " 83" x 182" |
| " 19 | 18 | " 8.28 " 62" x 180" |
| " 20 | 12 | Angles 2" x 4" x 14" x 22'-4" |
| " 21 | 12 | " 2" x 2½" x 2" x 22'-3" |
| " 22 | 1 | U.M. Plates ¼" x 18" x 15'-6" |
| " 23 | 1 | " " " ¾" x 34" x 16'-6" |
| " 24 | 1 | " 20.1" 40" Diam. |
| | | Shop Rivets |
| R 44 | 25 | Rivets ¾" x 2½" U.H. Cone Heads |
| | | Field Rivets |
| R 25 | 30 | Rivets ¾" x 2½" U.H. Cone Heads |
| " 26 | 60 | " " " " " " |
| " 27 | 60 | " " " " " " |
| " 28 | 120 | " " " " " " |
| " 29 | 1365 | " " " " " " |
| " 30 | 1120 | " " " " " " |
| " 31 | 2520 | " " " " " " |
| " 32 | 2456 | " " " " " " |
| " 33 | 810 | " " " " " " |
| " 34 | 90 | " " " " " " |
| " 35 | 100 | " " " " " " |
| " 36 | 80 | " " " " " " |
| " 37 | 2345 | " " " " " " |
| " 38 | 3900 | " " " " " " |
| " 39 | 70 | " " " " " Pan |
| " 40 | 60 | " " " " " " |
| " 41 | 900 | " " " " " " |
| " 42 | 3645 | " " " " " " |
| " 43 | 13940 | " " " " " " |

FIG. 216.—A 35,000-bbl. tank, 85 ft. in diameter by 35 ft. 2 in. in height.

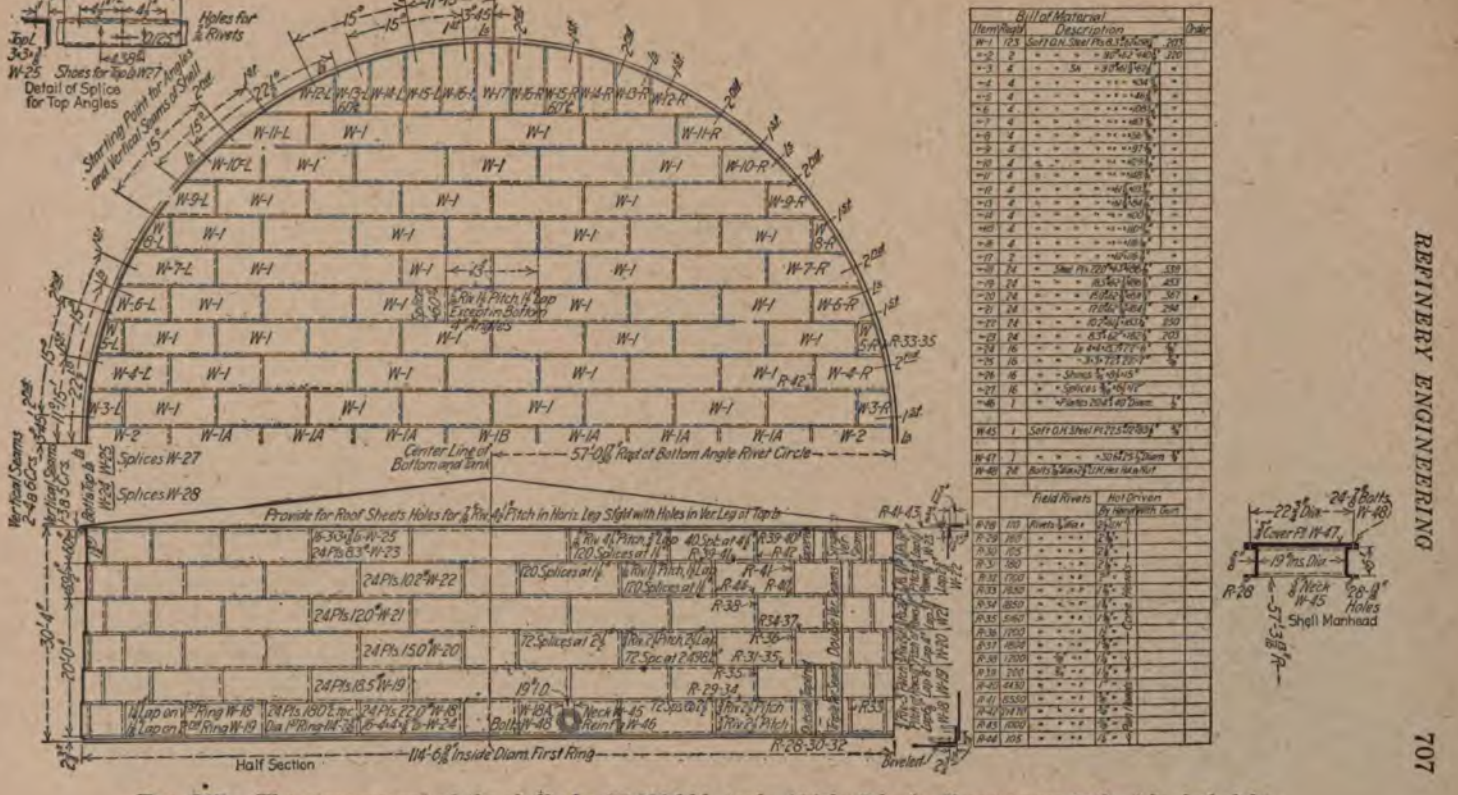



FIG. 217.—The arrangement of the shell of a 55,000-bbl. tank, 114 ft. 7 in. in diameter by 30 ft. 4 in. in height.



FIG. 218.—An oil storage tank of 55,000 bbl. capacity, built for the United States Navy, by the Treadwell Construction Company.



FIG. 219.—A crude oil storage tank of 55,000 bbl. capacity.



FIG. 220.—Structural steel framework for the steel roof of a storage tank.

C of circle = D × 3.14159.
D of circle = C × 0.3183.
Area of circle = $D^2 \times 0.7854$, also $C^2 \times 0.07958$.

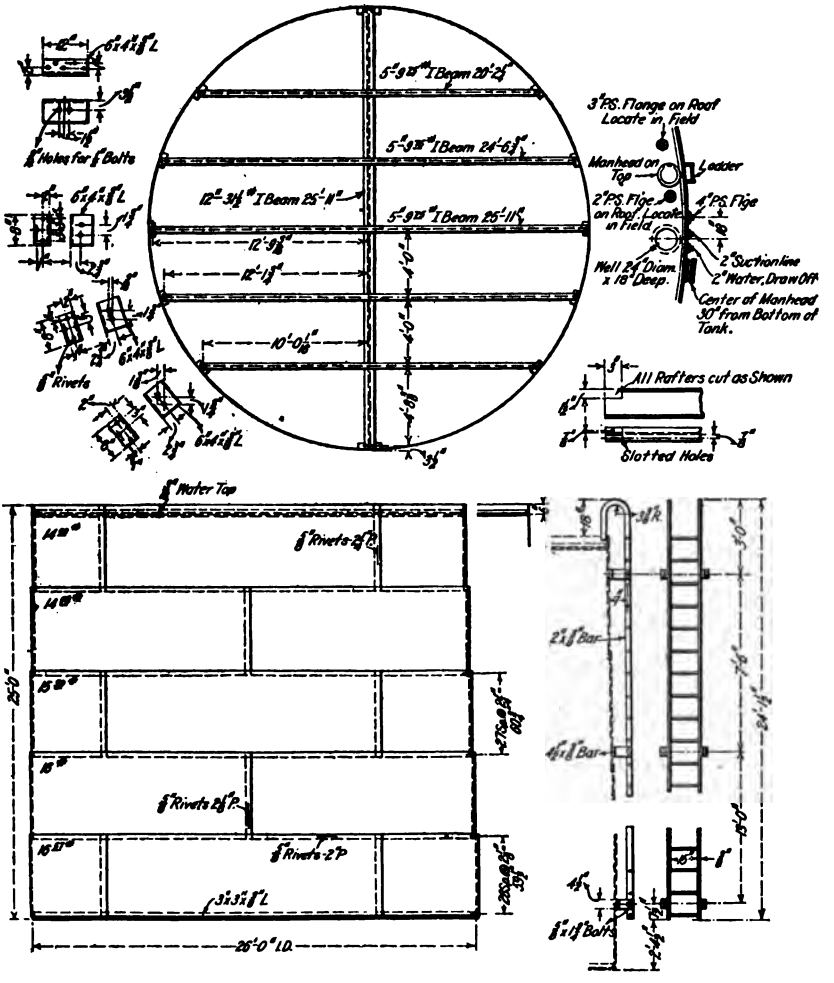



FIG. 221.—A water-top tank, 26 ft. in diameter by 25 ft. in height.

D of true sphere in inches cubed × 0.0022666 = U. S. G. in sphere.

U. S. G. at any inch in true sphere = $(3D - 2n)n^2 \times 0.0022666$, in which $n$ = height in inches, the diameter being also in inches. Where $n$ is more than ½D, compute capacity

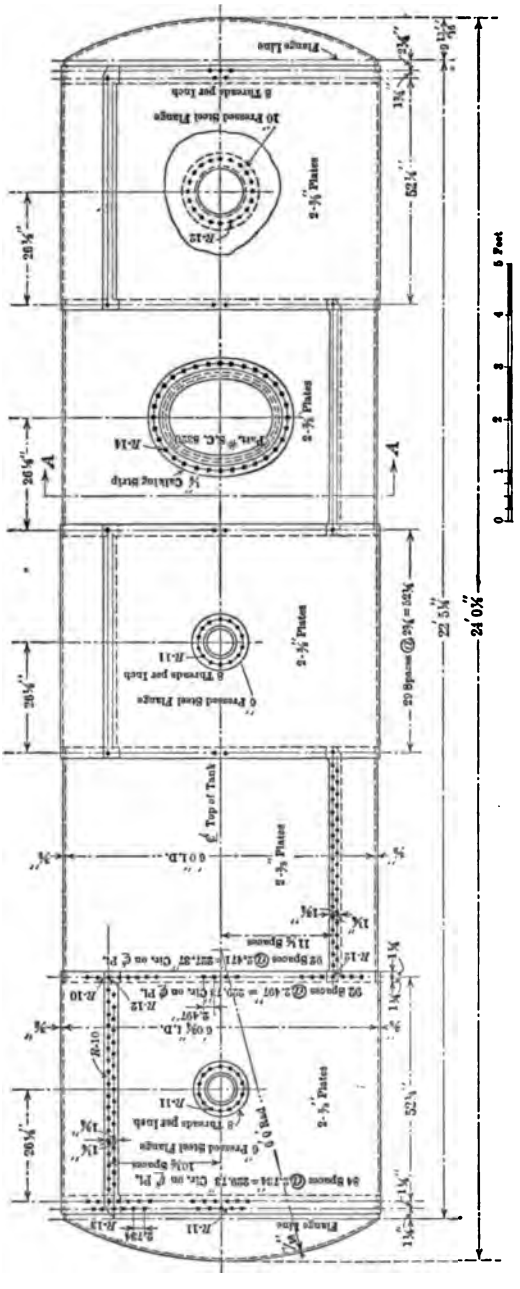



FIG. 222.—Details of a horizontal tank, 6 ft. in diameter by 24 ft. in length.

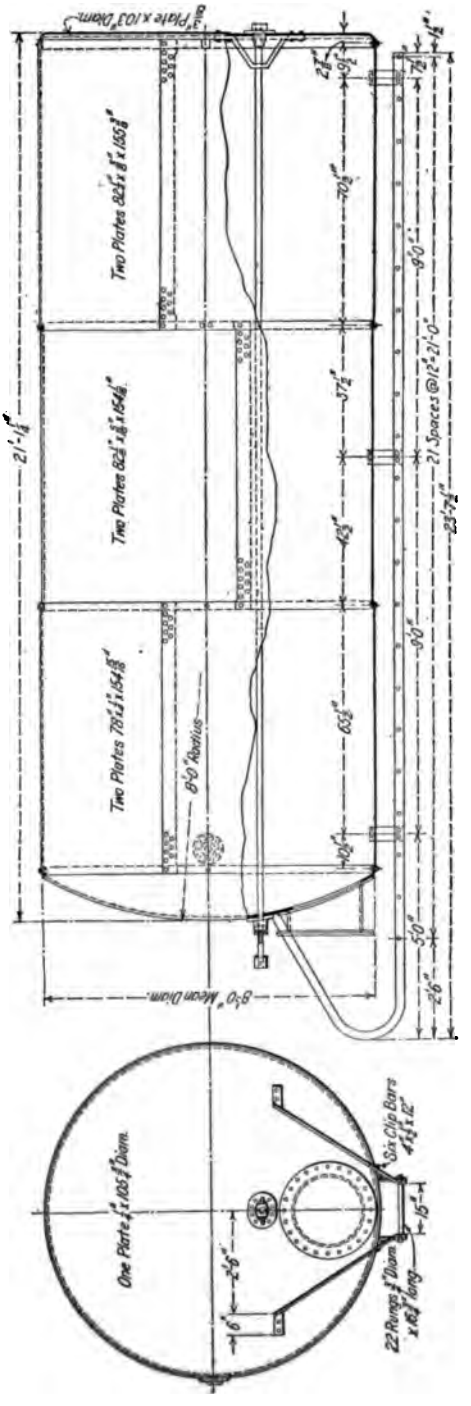



Fig. 223.—An acid storage tank, 8 ft. in diameter by 20 ft. in height.

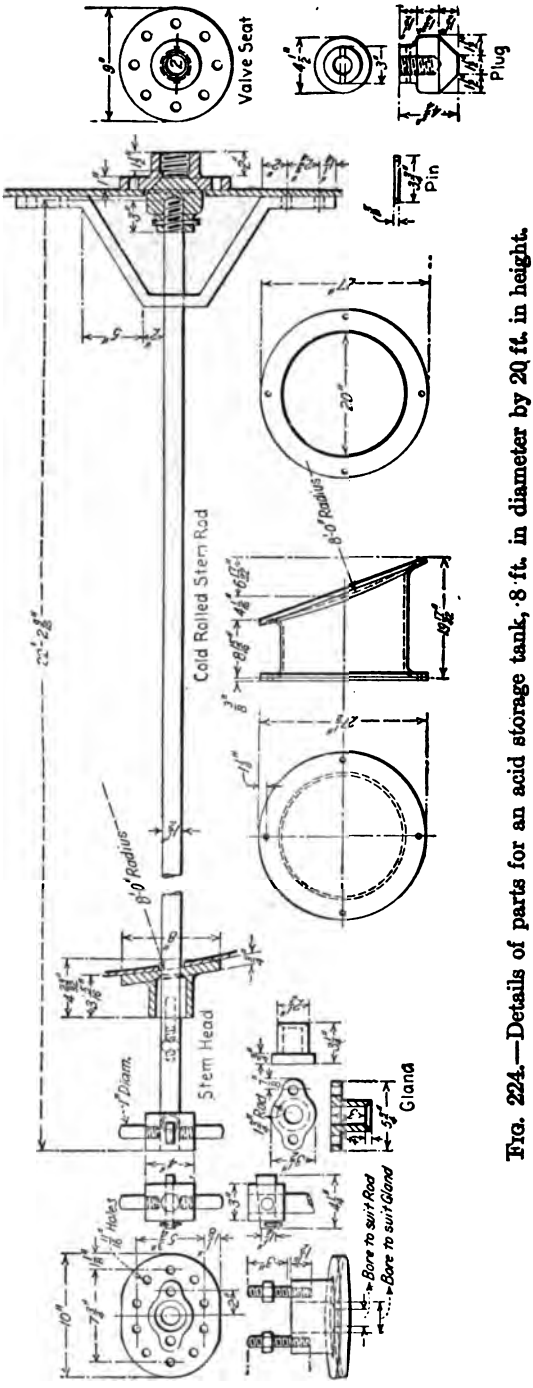



FIG. 224.—Details of parts for an acid storage tank, 8 ft. in diameter by 20 ft. in height.

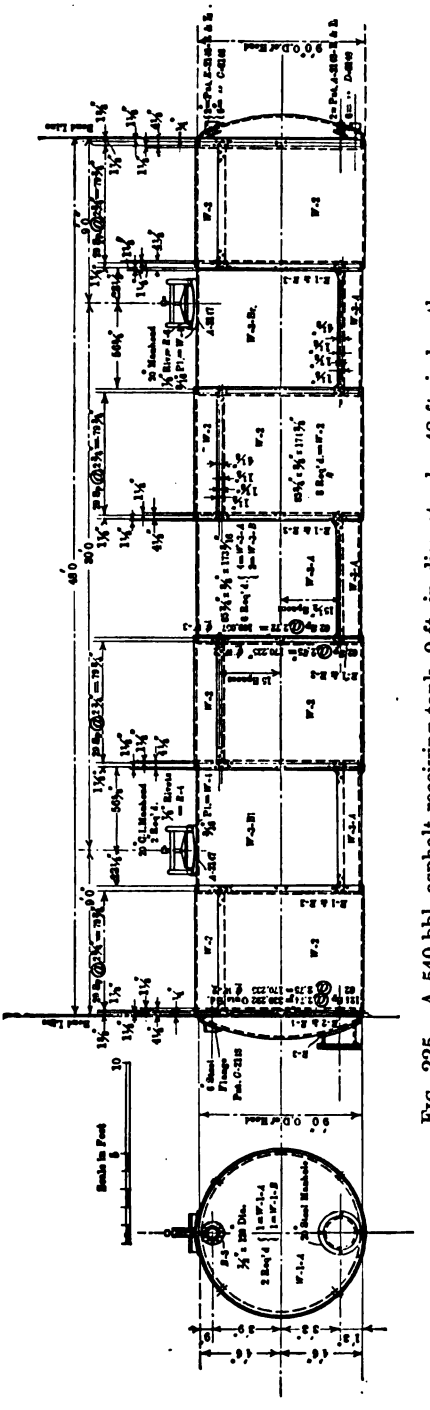


FIG. 225.—A 540-bbl. asphalt receiving tank, 9 ft. in diameter by 48 ft. in length.

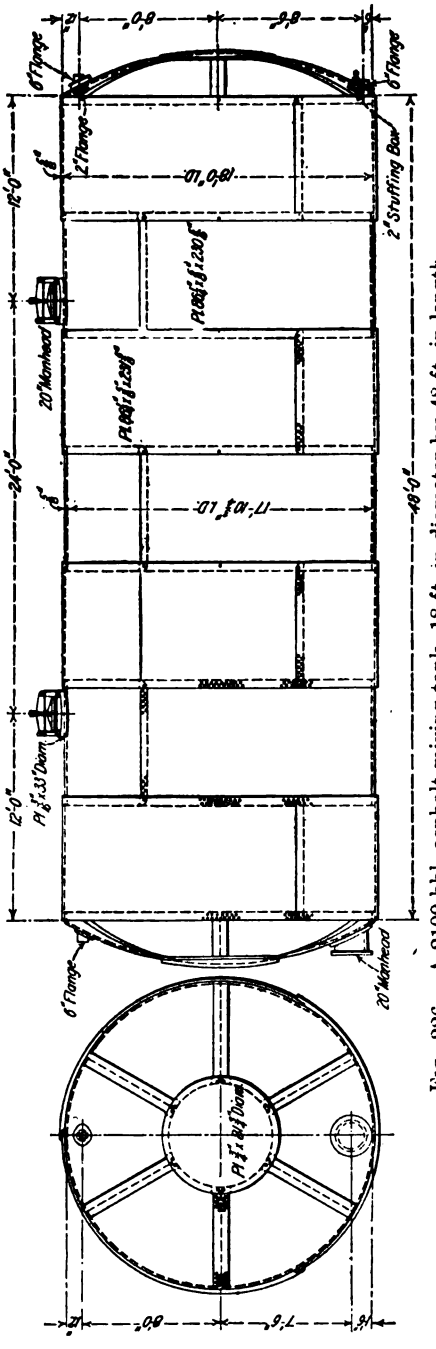



FIG. 226.—A 2100-bbl. asphalt mixing tank, 18 ft. in diameter by 48 ft. in length.

of sphere and deduct capacity of empty section, in which case the $n$ in above formula becomes inches of space.

To find internal circumference of tank, deduct from external



FIG. 227.—Receiving tanks, 9 ft. in diameter and 48 ft. in length.



FIG. 228.—Gauge-filling tanks at a California refinery.

circumference in feet 0.033 ft. for each $\frac{1}{16}$ in. thickness of iron and use internal circumference in making table.

Or more accurately: Find capacity per inch, using external circumference. Multiply external circumference in feet by 12;

multiply this by thickness of iron; divide result by 231 and deduct result from capacity per inch as found by using external circumference.

To get the smallest surface for a given capacity in a vertical cylindrical tank (assuming flat top), the height must equal the diameter.

The following formula shows how to ascertain the diameter (or height) for any capacity:

D = diameter of tank in feet;
H = height of tank in feet;
B = capacity in 40-gal. barrels;
$D = H = 1.895 \sqrt[3]{B}$.

Assuming 35 ft. the maximum height desirable, a tank of 6,304 bbl. capacity would be the largest which could be built by this rule.

**Painting.**—Many producers and marketers have adopted white or a light-colored paint for their crude petroleum and light oil storage tanks, mainly to minimize the losses through evaporation. It has been reported[1] that the evaporation from tanks painted white averages from 1 to 1.5 per cent. less than from red tanks and about 2.5 per cent. less than from tanks painted black. Hollow building tile are also being used for tankage insulation[2] by several western oil refiners.

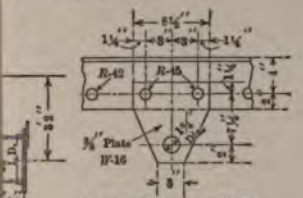
## STILLS

In the earlier American petroleum refineries, the stills, the capacity of which varied from 25 to 80 bbl., usually consisted of a vertical cylinder, constructed of cast or wrought iron, with a boiler plate bottom and a cast-iron dome, on which the "gooseneck" was bolted. In cast-iron stills the metal was objectionably thick. The charge was distilled almost to dryness, though the operation was not carried far enough to cause the residue to "coke." The method of treatment was, however, completely changed by the introduction of the "cracking process," and by the division of the distillation into two parts, one consisting in

[1] *Petrol. Gaz.*, **20** (1915), No. 4, 22. For a rather thorough consideration of the losses in the storage of crude oil, see GUISELIN, *Mat. grasses*, **4**, 2323, 2363 and 2403.

[2] "Adobe" coated with cement wash is a good heat insulator. Other cheap insulators are sawdust concrete and 1–10 cement-sand mortar.



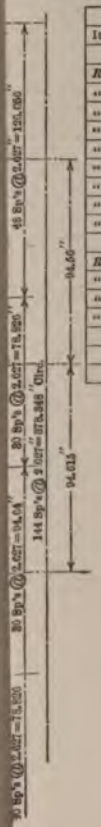

DETAIL OF TAR PLUG GUIDE



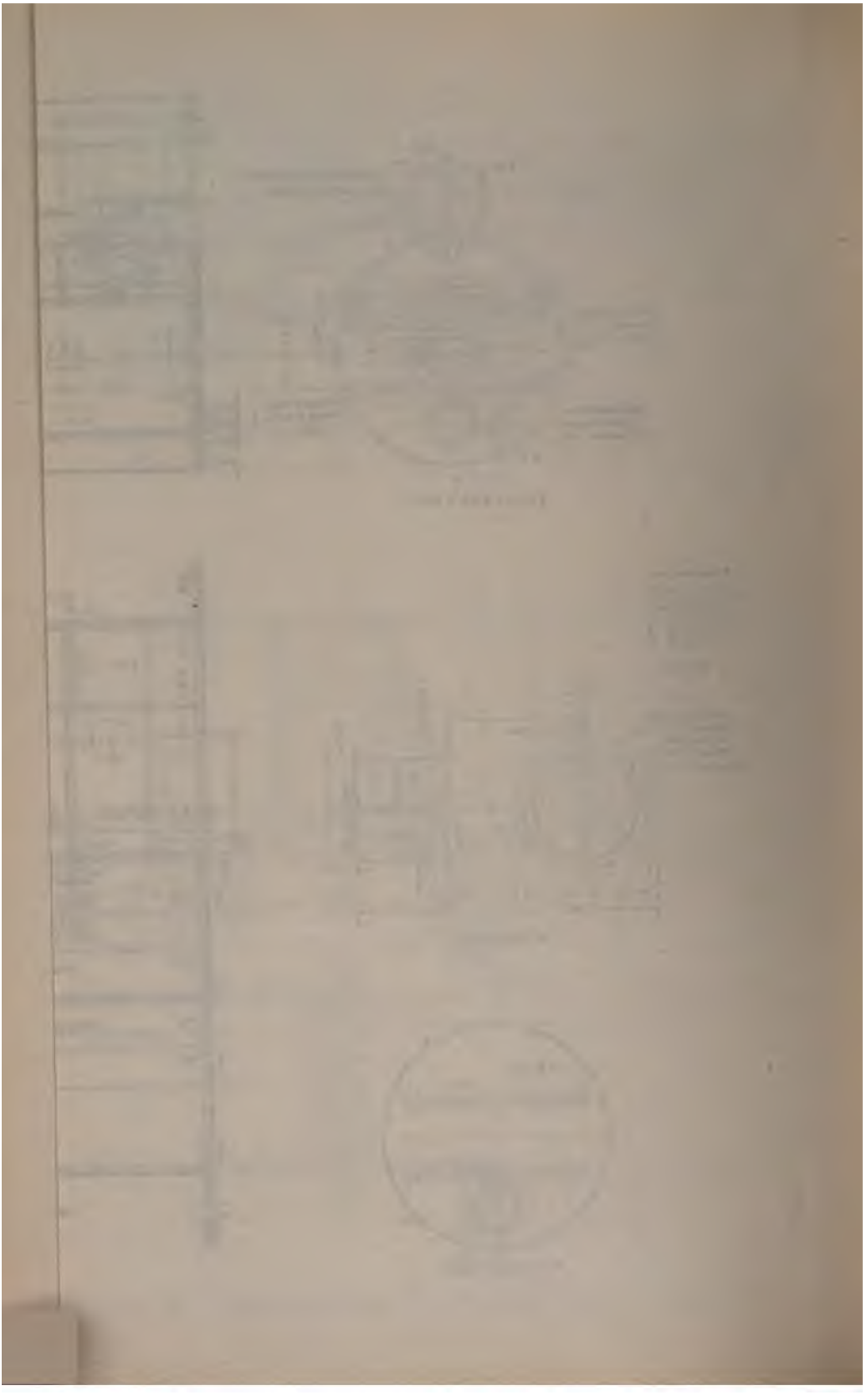

DETAIL OF FLANGE AND NIPPLE FOR TAR PLUG

| Bill of Material | | |
|---|---|---|
| Item | No | Description |
| | | Shop - Rivets - Std - Hds. - Cold Driven |
| R-40 | 1385 | Rivets ¾" Dia. x 1⅝" U.H. |
| " 41 | 1400 | " " " 1½" " |
| " 42 | 520 | " " " 1⅝" " |
| " 43 | 5 | " " " 1⅞" " |
| " 44 | 115 | " " " 1⅛" " |
| " 45 | 35 | " " " 2 " " |
| " 46 | 25 | " " " 2⅛" " |
| " 47 | 5 | " " " 2⅜" " |
| " 48 | 85 | " " " 2⅝" " |
| | | Field Rivets - Std. Heads Cold Driven |
| R-51 | 60 | Rivets ¾" Dia. x 1⅝" U.H. |
| " 53 | 25 | " " " 1⅞" " |
| " 54 | 5 | " " " 1⅛" " |
| " 57 | 280 | " " " 2¾" " |

| | | Bill of Material |
|---|---|---|
| W-No | Pieces | Description |
| W-1 | 1 | Plate 20.4# x 97⅜" x 241¼" |
| W-2 | 1 | " " " x 242¼" |
| W-3 | 2 | " 17.85# x 83½" x 166⅛" |
| W-4 | 2 | " " " x 155" |
| W-5 | 2 | " " " x 168¾" |
| W-6 | 4 | " 15.3# x 74½" x 133⅛" |
| W-7 | 3 | " " x 70½" x 133⅛" |
| W-8 | 2 | " 20.4# x 120" Dia |
| W-9 | 1 | " 15.3# x 41" x 117¼" |
| W-10 | 1 | " " x 44" Dia |
| W-11 | 8 | Angles ½" x 6" x 4" x 2' 6½" |
| W-12 | 2 | Plates 20.4# x 20" x 67½" |
| W-13 | 2 | " " x 29" Dia |
| W-14 | 2 | " " x 30" " |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | Bill of Material |
| W-15 | 1 | Bar ¾" x 4" x 5' 3 ¼" |
| W-16 | 1 | U.M. Pl. ⅜" x 8½" x 0' 10⅝" |
| W-17 | 2 | Plates 3/16" x 35" x 20" |
| W-18 | 1 | " 3/16" x 9" x 9" |
| W-19 | 1 | 1½" Pressed Steel Flanges Straight Thread |
| W-20 | 1 | 1½" " " " Tapered " |
| Stiffs C & D→ W-21 | 1 | 2" " " " " " |
| W-22 | 1 | 2⅛" " " " " " |
| W-23 | 2 | 3" " " " " " |
| W-24 | 1 | 3" " " " Straight " |
| W-25 | 1 | 4" " " " Tapered " |
| W-26 | 1 | 4" " " " Straight " |
| Stiff D→ W-27 | 2 | 6" " " " " " |
| " C-D W-28 | 2 | 10" " " " Tapered " |
| " B→ W-29 | 1 | 12" " " " " " |
| W-30 | 1 | Eye Bolts ¾"Φ x 2" to Inside of Eye ¾" Eye Hex. Nut |
| W-31 | 3 | " " " x 23" Long. ¾" Eye |
| W-32 | 48 | Bolts ¾" Φ x 2" Long. Square Shoulder Hex. Head. & Nut |
| W-33 | 1 | Std 4 Pipe Nipples 6" Long Thread both Ends |
| Stiff A-B→ W-34 | 1 | 12" Pressed Steel Flange Tapered Thread |
| " A-B-C→ W-35 | 2 | 8" " " " Straight " |
| | 2 | 22" C.I. Manheads |




(*Facing page* 716.)



the removal of the more volatile constituents of the oil, and the other in the distillation, usually in a separate still, of the residues from the first distillation, for the production of paraffin oils and wax.

At the present time, a horizontal underfired cylindrical still, having a charging capacity of from 200 to 1,200 bbl., is generally used in the distillation of crude oil; but what is known as the "cheese-box" still is even now employed by some refiners.

Figure 229 presents the detailed construction of a crude still,



FIG. 230.—Manner of assembling 1,000-bbl. crude stills.

10 ft. in diameter and 40 ft. in length; this still, which has a charging capacity of 500 bbl., is typical of those now in use in the Gulf and Mid-Continent fields. The dome at the top is connected with a vapor-pipe, or "goose-neck," that leads to the condenser.

Figure 230 shows the manner in which 1,000-bbl. crude stills, 16 ft. in diameter and 32 ft. in length, are assembled in the construction shops; they are afterward taken apart for shipment. Fig. 231 shows a 1,000-bbl. crude still in readiness for installation. When, however, the stills are of smaller diameter, say



FIG. 231.—A 1,000-bbl. crude still in readiness for installation.

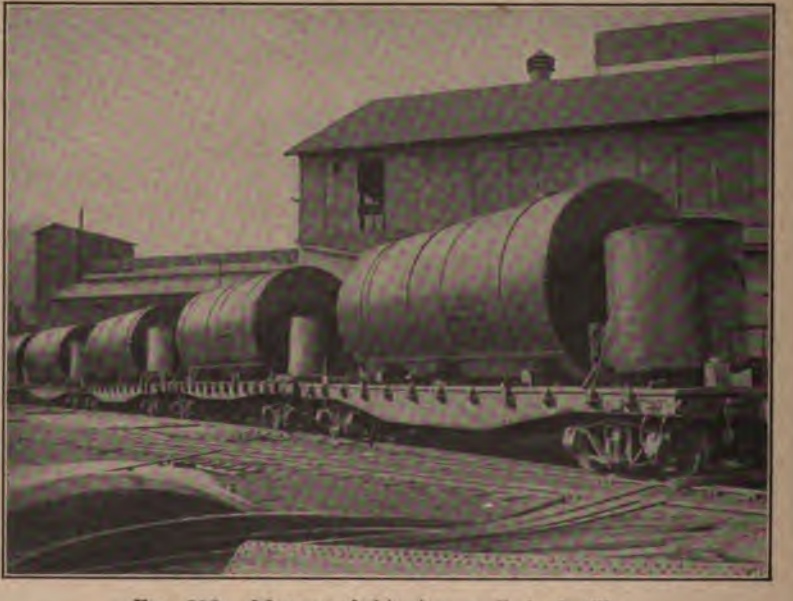


FIG. 232.—Manner of shipping small sized stills.

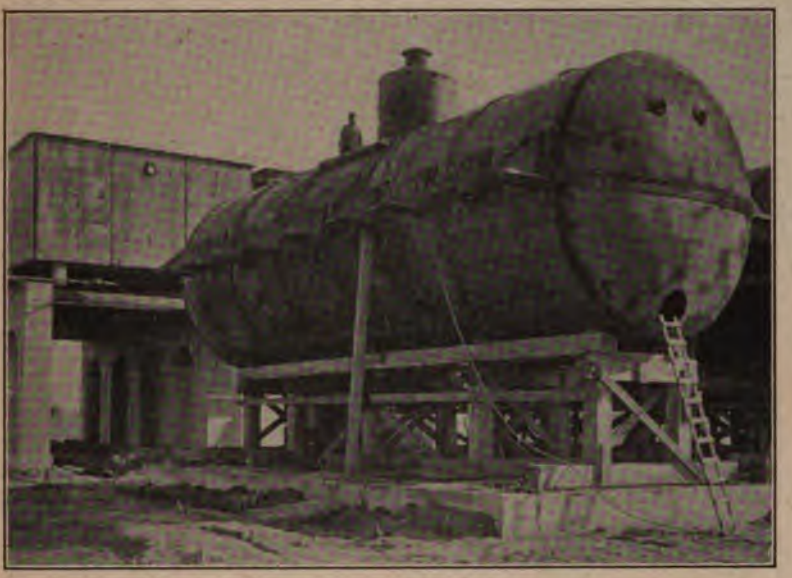


FIG. 233.—A still, 14 ft. 6 in. in diameter and 42 ft. in length, having a bottom of one plate.

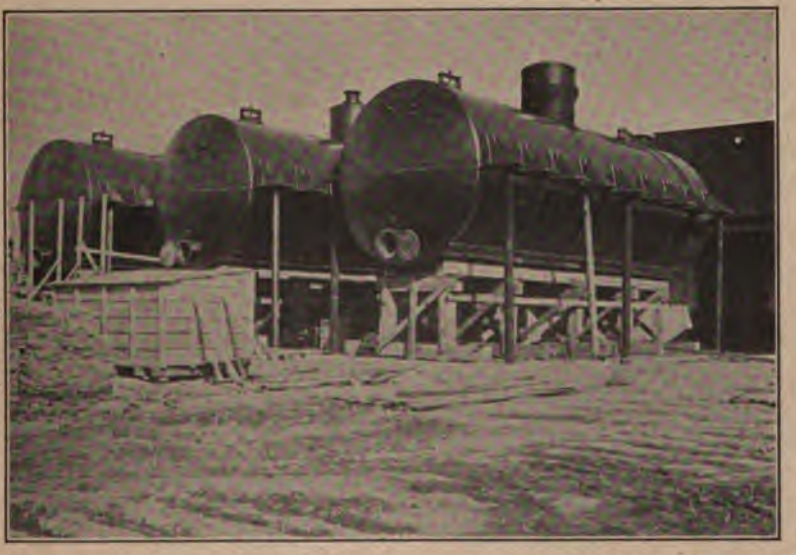


FIG. 234.—Three stills, 14 ft. 6 in. in diameter and 42 ft. in length, ready for installation at the refinery of the Huasteca Petroleum Company.



FIG. 235.—Rear of 10 ft. by 40 ft. crude stills in the course of installation.



FIG. 236.—A battery of 11 crude stills, each of 1,200 bbl. capacity, at the refinery of the Indian Refining Company, Laurenceville, Ill.



FIG. 237.—A "cheese-box" still of 150 bbl. capacity, 12 ft. in diameter by 8 ft. deep.



FIG. 238.—Stills of the "cheese-box" type in the Pennsylvania oil regions.

10 ft., they are built complete in the shops and then shipped intact, leaving off the domes to allow of clearance, as shown in Fig. 232. Fig. 233 illustrates a still, 14 ft. 6 in. in diameter by 42 ft. in length, the bottom of which is made of one plate, 10 ft. wide by 42 ft. long by ¾ in. thick; such stills have a weight of

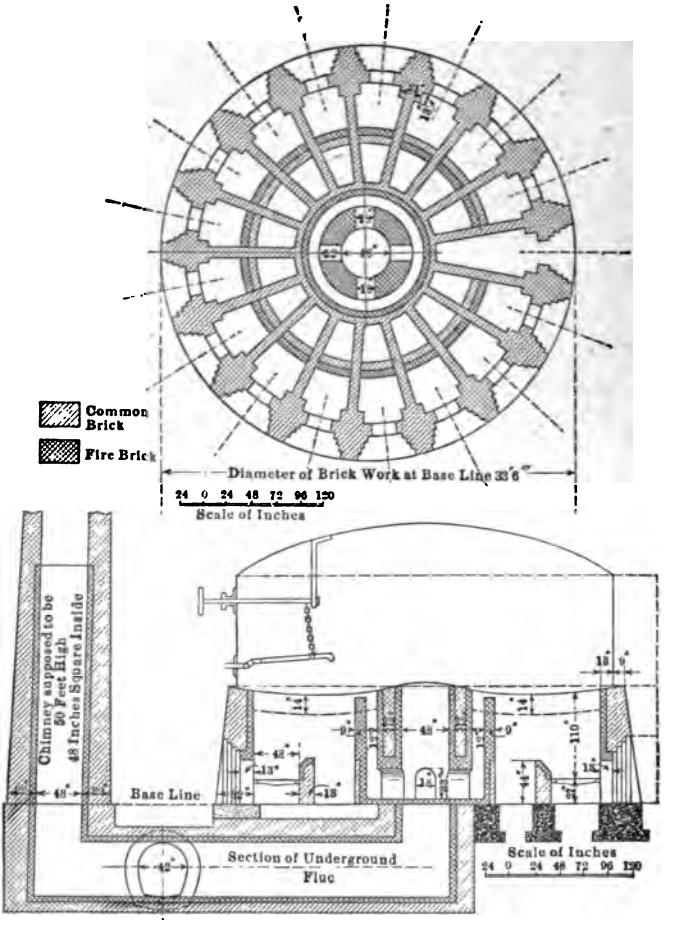



FIG. 239.—Method of setting "cheese-box" stills over a furnace.

84,500 lb. each. Three stills of the last-mentioned specifications are shown, ready for installation, in Fig. 234.

A battery of 11 crude stills, each of 1,200-bbl. capacity, at a large Illinois refinery, is presented in Fig. 236. It will be observed that in the battery shown the stills are completely bricked

in, so that the vapors shall be kept fully heated until they escape to the vapor-pipe; this is not, however, the general practice, and, especially because of the "cracking" process, the upper part is



FIG. 240.—The steam stills at the plant of the National Refining Company, Coffeyville, Kansas.



FIG. 241.—12 ft. by 35 ft. crude stills and a 14 ft. by 28 ft. steam still "cheese-box" type, under construction at Coffeyville, Kansas.

generally left exposed to the air. It may be noted here that horizontal cylindrical stills with internal flues[1] are used in

[1] The heating is effected by underfiring only, by underfiring aided by lateral flues, or by a combination of both the side-heating and the underfiring, with flues which pass through the still—an arrangement which is not employed in the United States.

the Baku field. These stills are set in batteries with a common wall between them, and a chimney common to all. They are usually heated by the burning of petroleum residues, and sometimes the distillation is conducted with the aid of superheated steam. The still dome usually has from five to seven pipes leading to the condenser, which is common to several stills. The stills are ordinarily placed in the open air, and the upper part is often not walled in, only tile coverings or a metal casing with a packing of glass-wool or slag being used.

The horizontal stills in American refineries are either end- or

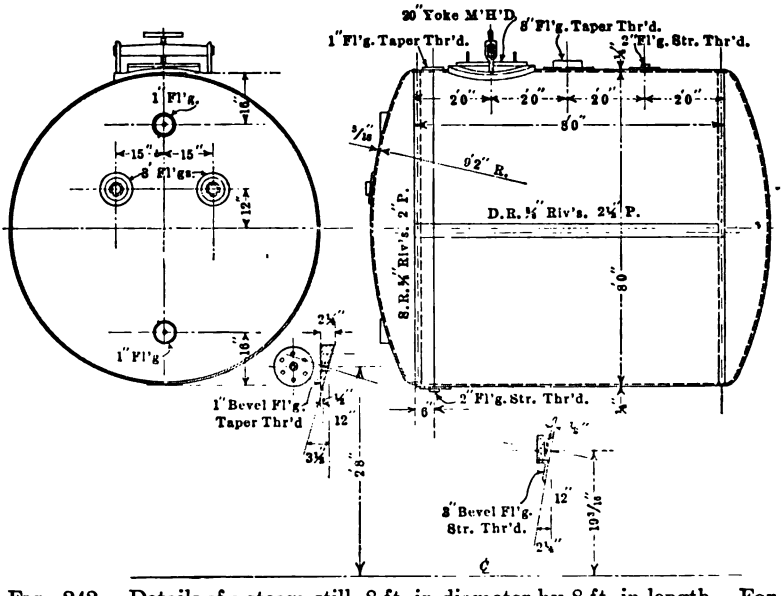



FIG. 242.—Details of a steam still, 8 ft. in diameter by 8 ft. in length. For specifications, see page 696.

side-fired. Side-firing is preferable because it gives a uniform distribution of heat. The fuel used is either coal or oil; in California, oil is in general use because of the scarcity of coal.

The so-called "cheese-box" still possesses a cylindrical body, and has a dome-shaped top constructed of iron plate and a steel-plate bottom. Formerly, this type was made in sizes up to 30 ft. in diameter by 9 ft. in depth, but at the present time the largest size of vertical still built for the Pennsylvania field is 16 ft. in diameter by 10 ft. in depth. A "cheese-box" still of 150 bbl. capacity, 12 ft. in diameter by 8 ft. deep, is shown in Fig.

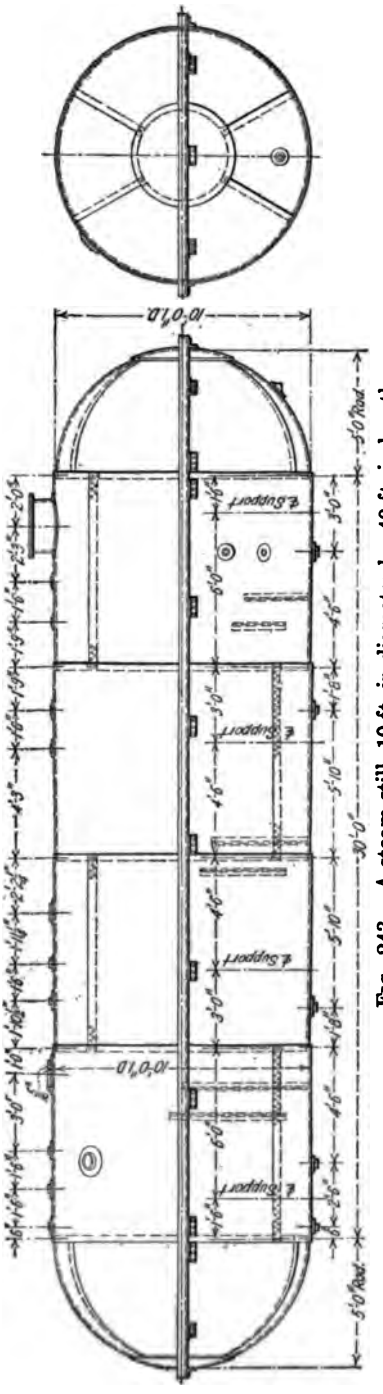



FIG. 243.—A steam still, 10 ft. in diameter by 40 ft. in length.

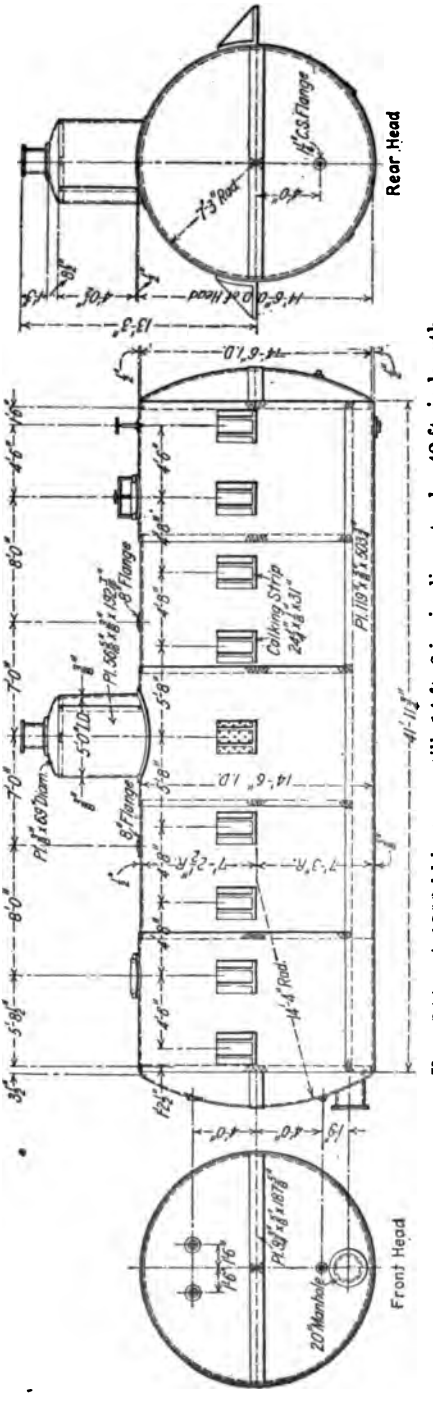



FIG. 244.—A 1200-bbl. steam still, 14 ft. 6 in. in diameter by 42 ft. in length.

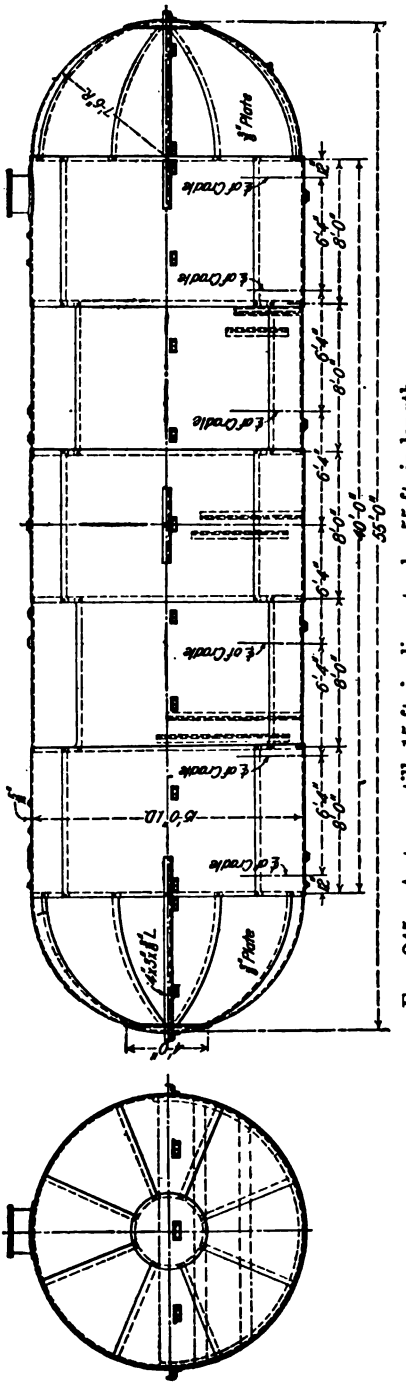



FIG. 246.—A steam still, 15 ft. in diameter by 55 ft. in length.

237. "Cheese-box" stills are set over a furnace on a series of brick arches (see Fig. 239). Vertical steam stills of large capacity are in use in the Kansas field (see Fig. 240).

The construction of three sizes of steam stills of the type most in use is shown in Figs. 242, 243, 244, and 245. Fig. 246 presents a finished steam still, 10 ft. in diameter by 40 ft. in length, such as is now in use in the Gulf and Mid-Continent fields; a still of this size weighs 21,500 lb. Fig. 247 gives the construction of a steam still tower, and in Fig. 248 are shown steam stills provided with 6-ft. towers containing four baffle-plates each.



FIG. 246.—A steam still, 10 ft. in diameter and 40 ft. in length, of the type in use by the Gulf Refining Company.

The term "tower still"[1] is applied to any still which is provided with tower-like aerial condensers[2] connecting with the vapor-pipe, or "goose-neck," and interposed between the stills and water-cooled condensers. The towers generally consist of a top and bottom gas chamber connected by pipes, around which air circulates, thus producing partial condensation. The goose-neck carrying the hot vapors from the stills connects into the bottom gas chamber of the first tower and passes up through

[1] See p. 456.

[2] These towers act, in fact, as fractional condensers.

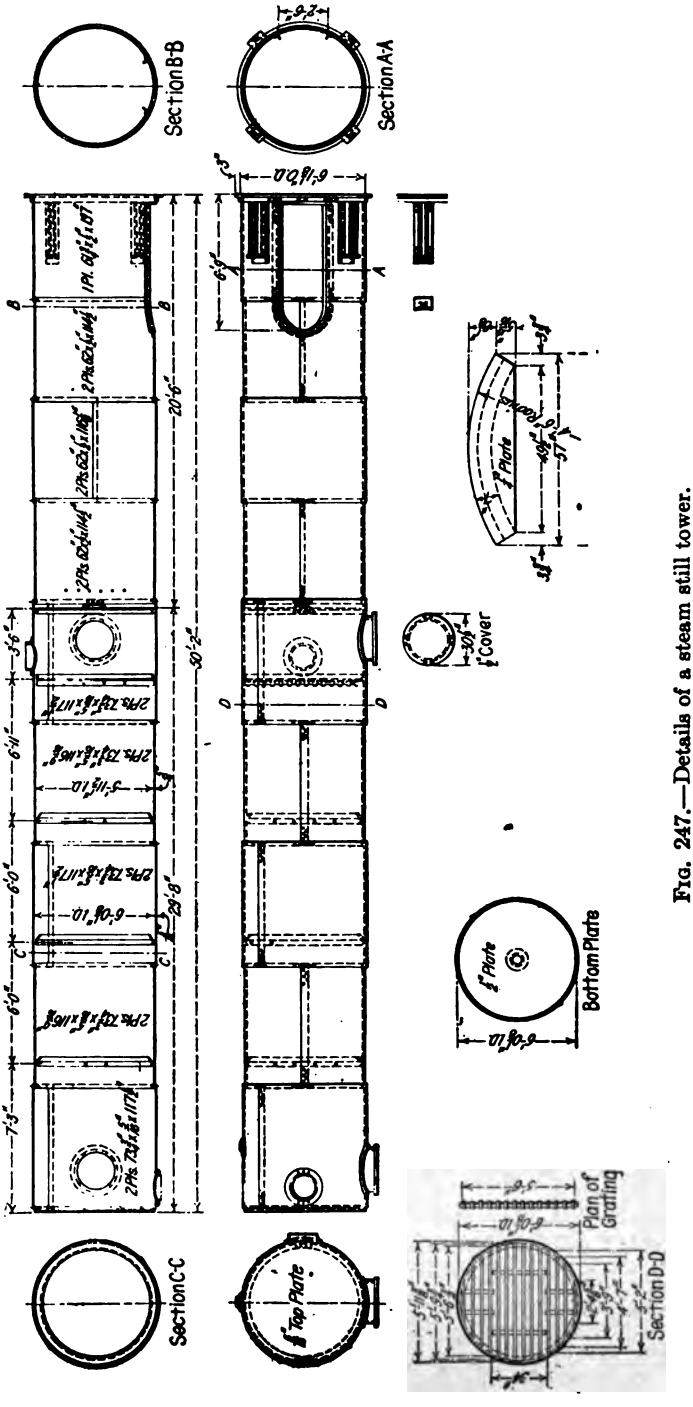



FIG. 247.—Details of a steam still tower.



FIG. 248.—Steam stills provided with 6-ft. towers.

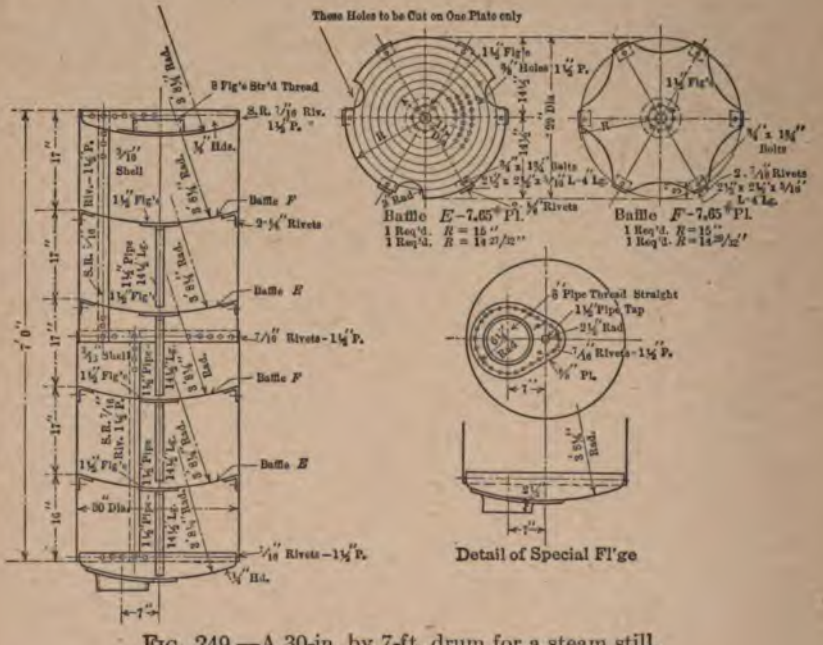



FIG. 249.—A 30-in. by 7-ft. drum for a steam still.

the pipes through the top gas chamber into the bottom of the second tower, and so on through the series to an ordinary water-cooled condenser.[1]

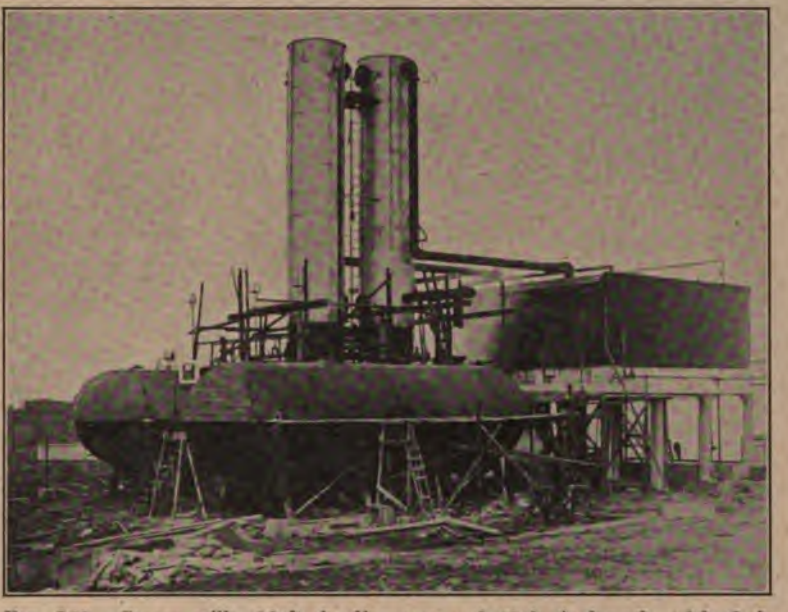


FIG. 250.—Steam stills, 10 ft. in diameter and 40 ft. in length, with their towers and condenser boxes, under construction at a California refinery.



FIG. 251.—Dephlegmator for interposition between the dome of a still and a condenser.

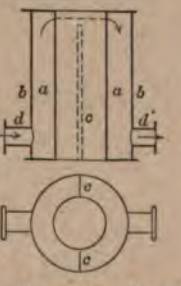
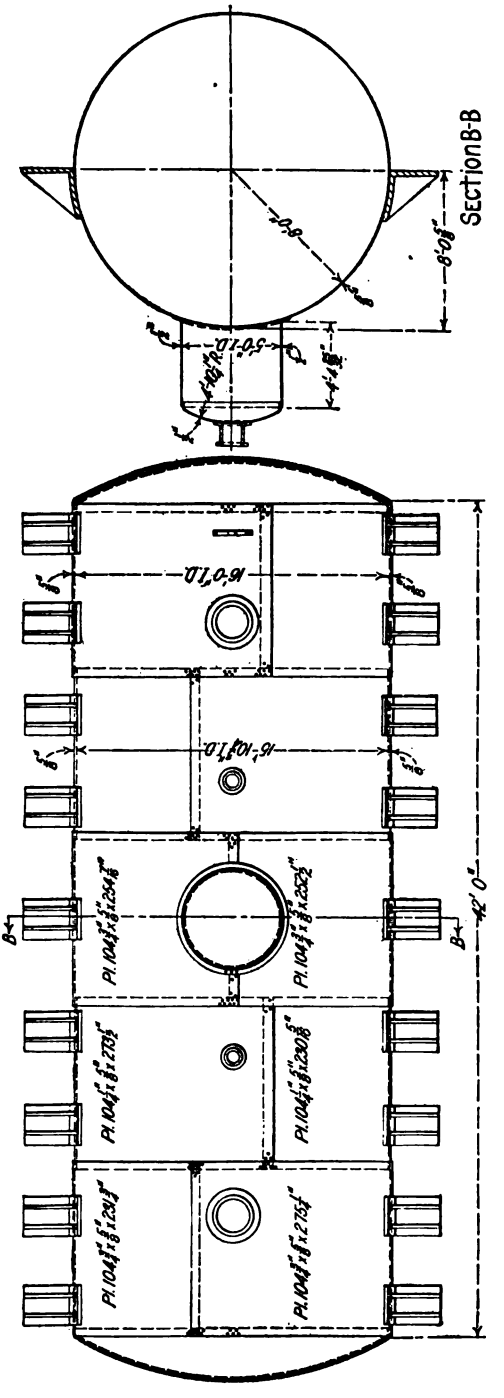



FIG. 252.—Separator for arresting any oil which is mechanically carried over from a still by the vapor.

[1] T. T. GRAY in ROGERS' "Industrial Chemistry," 2d ed., 505. On towers, see also C. R. EWING, United States Patent 1083998, Jan. 13, **1914**; W. C. KOEHLER and L. LINK, United States Patent 1084016, Jan. 13, **1914**; and G. H. GILLONS, United States Patent 1084080, Jan. 13, **1914**.


Section B-B
42' 0"
B
B

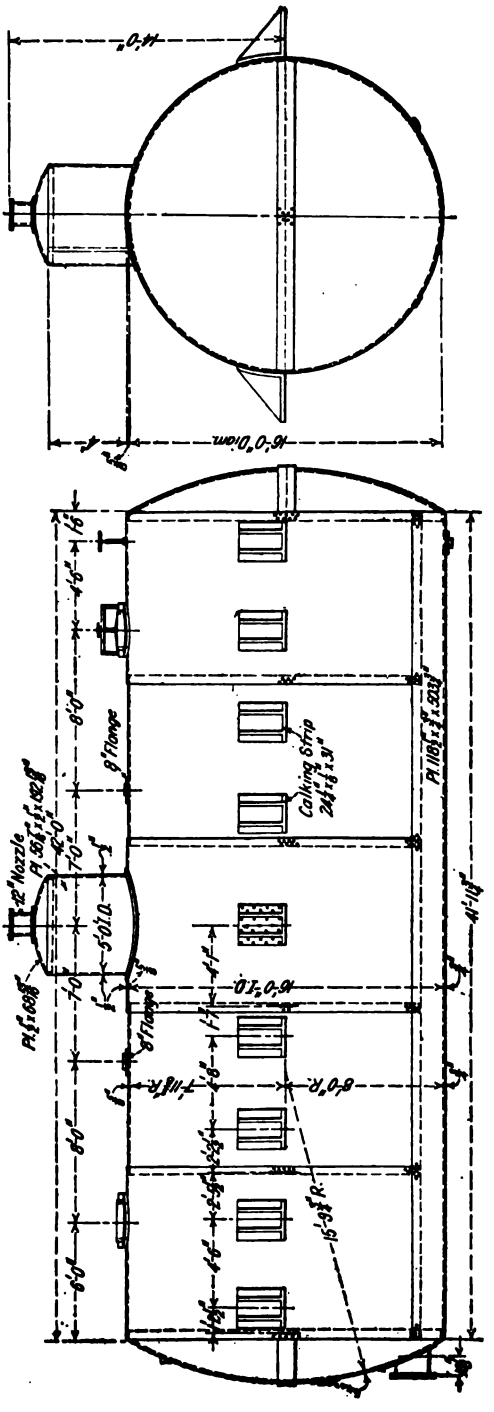



FIG. 253.—1500-bbl. blowing still, 16 ft. in diameter by 42 ft. in length.

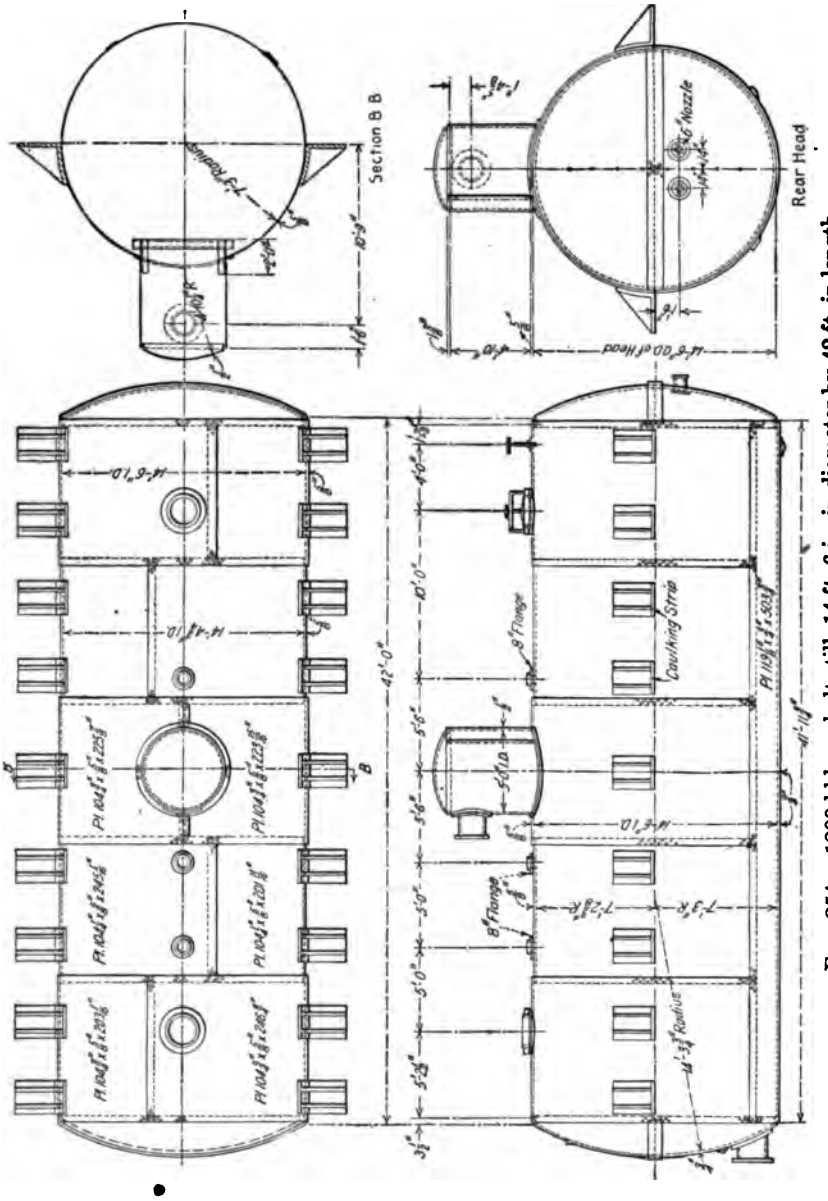



FIG. 254.—1200-bbl. asphalt still, 14 ft. 6 in. in diameter by 42 ft. in length.

Tower stills are said to possess a number of advantages over the older types. Since condensation occurs in each tower, what would otherwise be one product is separated into as many products as there are towers; consequently further necessary redistillation is reduced to a minimum.

Upright stills have been introduced for the distillation of benzine, but these are disadvantageous. The available heating surface decreases as the contents of the still diminish in volume, and is really smallest when the most volatile portions of the benzine have been expelled and the highest temperature is required.[1]

For the purpose of arresting any oil mechanically carried over by the vapor, a dephlegmator[2] of the form shown in Fig. 251 has been interposed between the dome of the still and the condenser. In the separator for this purpose shown in Fig. 252, *a* is an iron cylinder surrounded by a jacket, *b*, the space between them being divided, except at the top, by plates, *c*. The vapor enters at *d*, and passing through the apertures above the baffle-plates, *c*, as indicated by the arrow, escapes through $d^1$ to the condenser. The separated oil is returned to the still by a pipe.

A 1,500-bbl. blowing still, 16 ft. in diameter by 42 ft. in length, is exhibited in Fig. 253. Fig. 254 presents the details of a 1,200-bbl. asphalt still, 14 ft. 6 in. in diameter by 42 ft. in length.

The life of a still is dependent upon the treatment it receives at the hands of the workmen, the nature of the oil run, how far the distillation is carried, and the quality of the iron used in the construction.[3]

## CONDENSERS

Condensers are employed in the refinery to cool the vapors of petroleum distillates to temperatures below those at which these vapors are converted into the liquid state under the pressure which obtains. The condensation of such vapors is generally effected by bringing them into contact with a cold surface, to which they impart their heat, suitable arrangements being usually

[1] Veith, *Dingler's polyt. J.*, **282** (1891), 159.

[2] On the dephlegmator in oil refineries, see Berg, *Petrol. Rev.*, **4** (1901), 553.

[3] Small cracks in a still may be repaired by bolting on a patch of wrought iron, making a tight joint by the use of a mixture of manganese dioxide and oil; but, better still, by autogenous welding. On the corrosion of tar stills, see Warnes and Davey, *J. Soc. Chem. Ind.*, **29** (1910), 657.

made for conveying this heat away from the cooling surface. Air cooling is occasionally depended upon for this latter purpose, but the usual arrangement consists in causing water to circulate over the exterior of the condensing apparatus.

Some of the factors involved in the design of efficient condensers are the following:

1. The cooling surface should be constructed of material upon which the vapor has no chemical action and which is resistant to corrosion,[1] and should be capable of withstanding repeated variations of temperature without fracture. For practical purposes, this material must, of course, be of reasonable cost.

2. The cooling surface area should be as large as possible, without making the apparatus unduly extensive and heavy.

3. The material of construction should have a high thermal conductivity.

4. The design should be such that constituent parts of the apparatus may be removed for cleaning or replacement.

While it is not possible to obtain materials entirely satisfactory in each of the above respects, it has been found that materials are available which combine cheapness with reasonable satisfaction in the foregoing particulars. Steel or cast iron is the material which comes nearest to meeting all requirements in practice.

Many different types of condensers have been used in the petroleum industry, the best known forms being those in which the vapors traverse coiled or parallel iron pipes of large cooling surface. Water has been generally adopted as the cooling medium, although in such operations as the production of high boiling-point oils the exposure of the condensing surface to the air has been found to serve.[2] It is sometimes necessary to keep the condensing water warm, or even almost boiling, in order to prevent the solidification of paraffin in the pipes; in fact, this practice is general in running off the wax distillate from Ohio and Pennsylvania petroleum.

The quantity of water required to effect the condensation of oil vapors and to cool the distillate to a desired point, may be

[1] The corrosion of condenser pipes has been caused by the action of hydrochloric acid, formed from the chlorides in the crude oil.

[2] A very large condensing surface is, however, required in air condensation. VEITH ("Das Erdöl," 141) states that condensation by air requires almost 200 times the condensing surface which suffices when water is employed.

calculated by the aid of the specific heat and latent heat of evaporation of the particular product to be distilled. For example, if

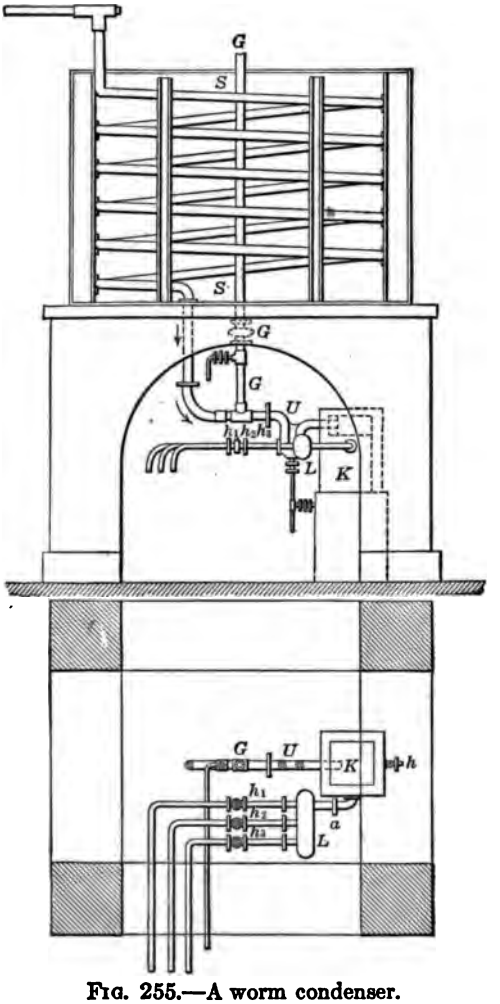



FIG. 255.—A worm condenser.

The condensed liquid passes into a U-tube, *U*, forming a seal, which prevents the passage of uncondensed gas, and diverts it into an escape-pipe, *G*. The liquid passes into a tank, *K*, from which the condensed water, which separates, is drawn off from time to time through a tap, *h*. The oil flows through a pipe, *a*, and a separator, *L*, consisting of a cast-iron pipe, with several branches connected through taps, $h_1$, $h_2$, $h_3$, etc., with pipes which lead to storage-tanks. The tank *K* is fitted with a window so that the color of the distillate may be observed and the distribution of the products controlled, the different qualities being directed into their respective tanks by opening the proper cocks. Taps are fitted to the bottoms of the U-tube and of the tank *K*, for running-off sediment.

1,000 Imp. gal. of a product having a specific gravity of 0.756 are to be distilled, and the distillation is to be conducted at the rate

of 100 gal. per hour, then 1,000,260 B.t.u. 7,560 lb. of product × 133.5°F., the latent heat of evaporation of the fraction having a specific gravity of 0.756, must be removed from the vapors in order to condense them to a liquid having a temperature of 175°F., the boiling point of the fraction. If the condensate is to be cooled to 60°F., 443,741 thermal units [7,560 × 175°–60° × 0.5104, the specific heat] must also be removed. Since the distillation is to require 10 hr., the number of thermal units per hour to be absorbed by the condenser will amount to 145,300 [(1,000,260 + 443,741) ÷ 10]. Assuming that the initial tem-

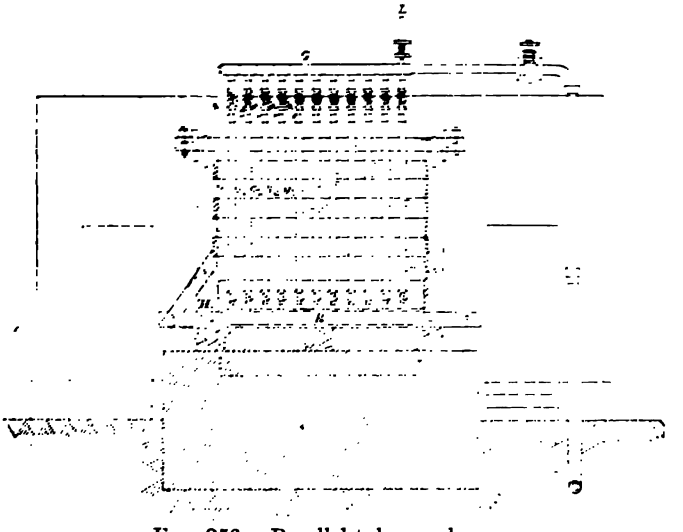
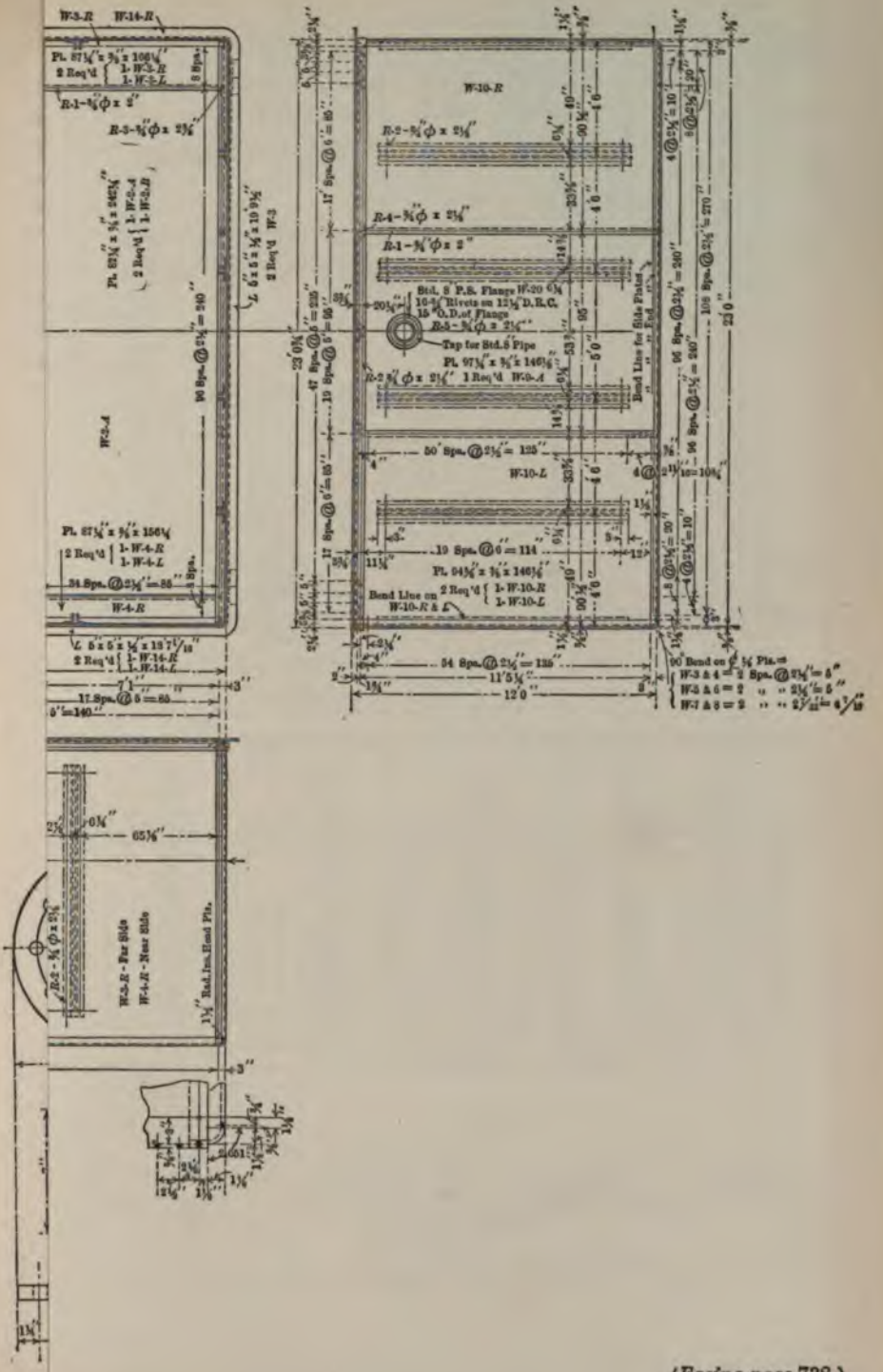


FIG. 256.—Parallel-tube condenser.

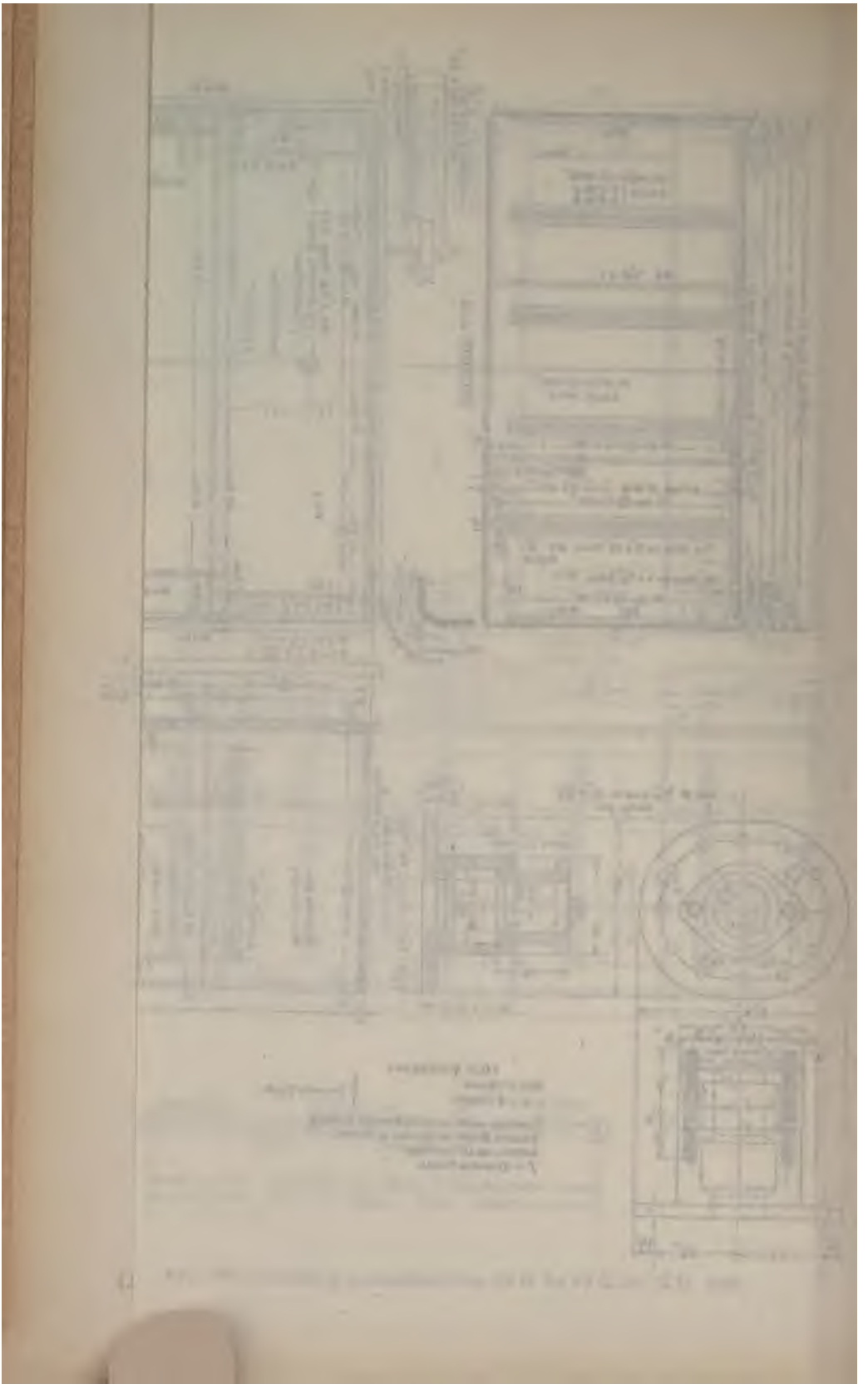
The distillates flow through a syphon into a series of parallel troughs, *R*, in communication with a stop-cock, *H*; while the uncondensable gas, which is arrested by the syphon, passes up the vertical pipes, *g*, etc., through the cocks, *h*, etc., into the horizontal pipe, *G*, whence it is conducted into a gasometer, or is allowed to escape into the air at *L*.

perature of the condensing water is 50°F. and that the temperature of the overflow will be 80°F., each pound of water will therefore absorb 30 thermal units. As 145,300 units are to be absorbed, 4,843 lb. (145,300 ÷ 30) or about 485 Imp. gal. of water per hour will be required. In this way the approximate quantity of water required can be determined, but in practice 10 per cent. should always be added to the result.[1]

[1] I. I. REDWOOD'S "Mineral Oils and their By-Products," **1914**, 202. On the latent heats of evaporation of petroleum and its products, see p. 108. On specific heats, see p. 104.




(*Facing page 738.*)



While the worm condenser is in general use, the spiral form used in the early refineries has been replaced by a series of straight tubes connected by elbows and passing across or along the sides of a tank or condenser box made of iron, or by other arrangements, through which the water circulates from below upward. The extent of the condensing surface varies with the size of the still. Veith[1] states that a still of 500- to 700-bbl. capacity should have a series of condensing tubes of a total length of 984 ft. 3 in and an average section of 6 in. The pipes may diminish in diameter from 8 to 10 in. where the vapors enter, to 2 to 3 in.

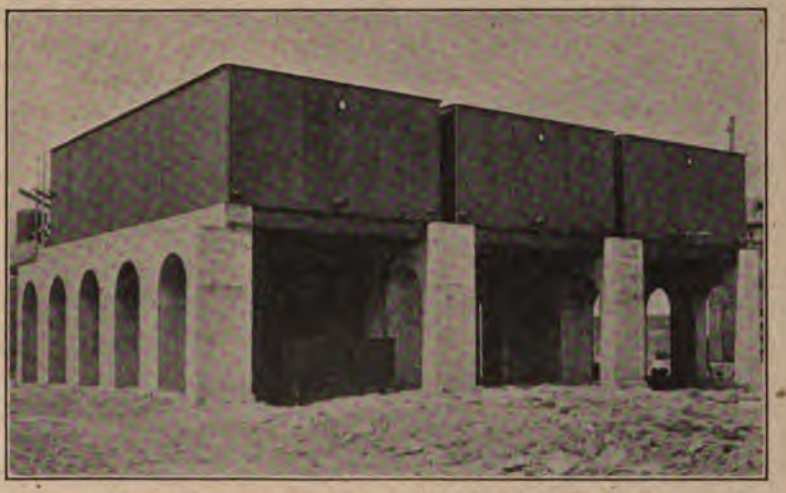


FIG. 258.—Condenser boxes, 23 ft. by 48 ft. by 12 ft.

where the condensed product escapes. The condensing capacity should always be sufficient to allow of the still being run at a maximum rate without occasioning a loss of vapors owing to imperfect condensation. According to Scottish shale-oil practice, there should be 1 sq. ft. of condensing surface for each gallon of oil distilled per hour for heavy oils; 1¼ sq. ft. for illuminating oils; and 1¾ to 2 sq. ft. for gasoline and naphtha. I. I. Redwood[2] has expressed the opinion that the internal area of the cross-section of the condenser worm at the inlet to the condenser should be 0.05 sq. in. per gallon of distillate per hour, and that this size should be continued for about one-third of the total length, then

[1] *Op. cit.*, 142.
[2] *Op. cit.*, 199.

reduced by ½ in. for the next third and by another ½ in. for the last third.

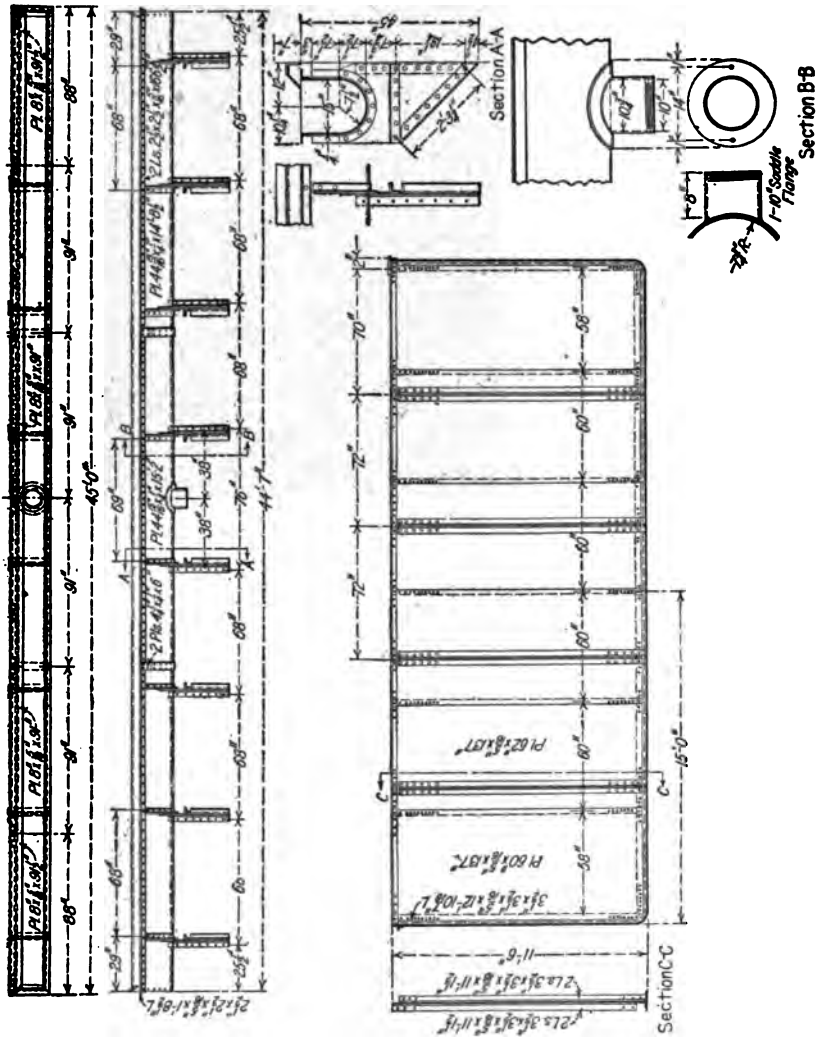
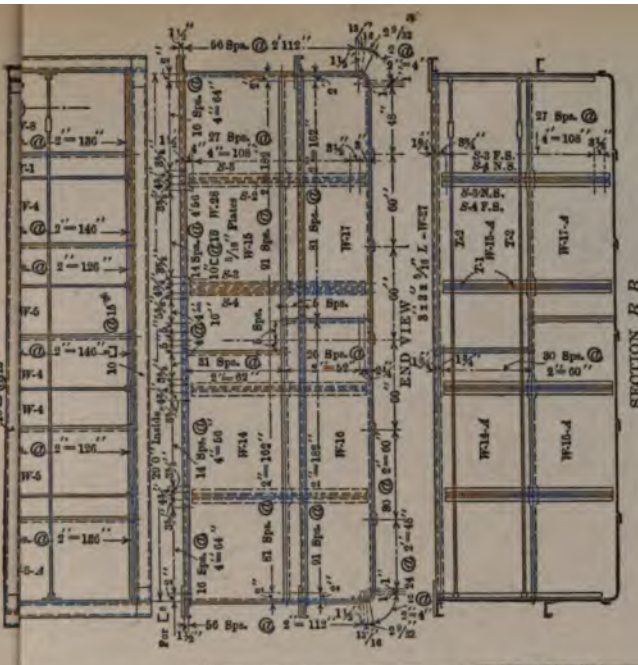
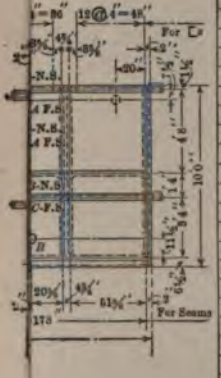


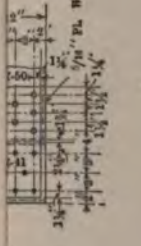

FIG. 259.—Overflow box and diaphragm for a condenser box, 30 ft. by 45 ft. by 11 ft. 6 in.

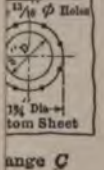
An ordinary worm condenser, fitted with an arrangement for separating the distillates,[1] is shown in Fig. 255; and Fig. 256

[1] A similar cast-iron tube condenser has been described by ENGLER, *Dingler's polyt. J.*, **260**, 438.







## Bill of Material

| Item No. | No. Req'd | Description | Item No. | No. Req'd | Description |
|---|---|---|---|---|---|
| W 1 | 24 | Plates 12·75# x 62⅝" x 160¾" | | | |
| " 2 | 2 | " " " " x 168¾" | | | Field Rivets R.C. Std. |
| " 3 | 2 | " " " " x 188¾" | | | R.C. Make Cold Driven |
| " 4 | 2 | " " " " x 148¾" | R-40 | 200 | Rivets ⅝" Dia. x 1" U.H: Std. Hd. |
| " 5 | 2 | " " " " x 128¾" | " 41 | 10020 | " " " 1⅛" " " " |
| " 6 | 12 | " " " 61⅜" x 160¾" | " 42 | 1480 | " " " 1⅜" " " " |
| " 7 | 2 | " " " " x 178¾" | " 43 | 300 | " " " 1⅝" " " " |
| " 8 | 2 | " " " " x 138¾" | " 44 | 2060 | " " " 1⅞" " " " |
| " 9 | 24 | " " " 58¾" x 160¾" | " 45 | 220 | " " " 2⅝" " " " |
| " 10 | 2 | " " " " x 146¾" | " 46 | 140 | " " " 1 Over Hd. Csk. Hd. |
| " 11 | 2 | " " " " x 136¾" | | | |
| " 12 | 2 | " " " " x 170¾" | | | |
| " 13 | 2 | " " " " x 180¾" | | | |
| " 14 | 9 | " " " 64¾" x 168⅜" | | | Shop Rivets R.C. Std. Hot Driven |
| " 15 | 9 | " " " " x 188⅜" | R-50 | 240 | Rivets ⅝ Dia. x 1⅜" Lg. U.H: Std. Hd |
| " 16 | 9 | " " " 61" x 188⅜" | | | |
| " 17 | 9 | " " " " x 168⅜" | | | |
| " 18 | 1 | U.M.Pl. 5/16 x 8⅜" x 12'0" | | | |
| " 19 | 1 | " " " x 12 x 11'10" | | | |
| " 20 | 2 | " " " x 11⅜" x 12'1¾" | | | |
| " 21 | 1 | " " " x 8⅜" x 11'10" | | | |
| " 22 | 12 | Bars ⅛ Dia. x 24'2" | | | |
| " 23 | 32 | " " " x 27'4⅜" | | | |
| " 24 | 64 | " " " x 11'6⅜" | | | |
| " 25 | 64 | Angles 5/16" x 3½" x 3" x 9"4⅜" | | | |
| " 26 | 128 | " " " x " x 9'6⅜" | | | |
| " 27 | 7 | " " " x " x 28'11" | | | |
| " 28 | 4 | Channels 10" x 15# x 29'0" | | | |
| " 29 | 4 | " " x " x 20'6" | | | |
| " 30 | 4 | " " x " x 21'10" | | | |
| " 30A | 4 | " " x " x 21'8" | | | |
| " 32 | 96 | ⅛" Turnbuckles | | | |
| " 33 | 192 | Bolts ⅛" Dia. x 3" Hex. Hd. & Nut | | | |
| " 34 | 8 | 6 Pressed Steel Flanges ½" Pl. | | | |
| " 31 | 4 | Channels 10" @ 15# x 20'8" | | | |
| " 35 | 4 | " " " " x 22'4" | | | |
| | 12 | 3 Pressed Steel Figs St. Thd | | | |

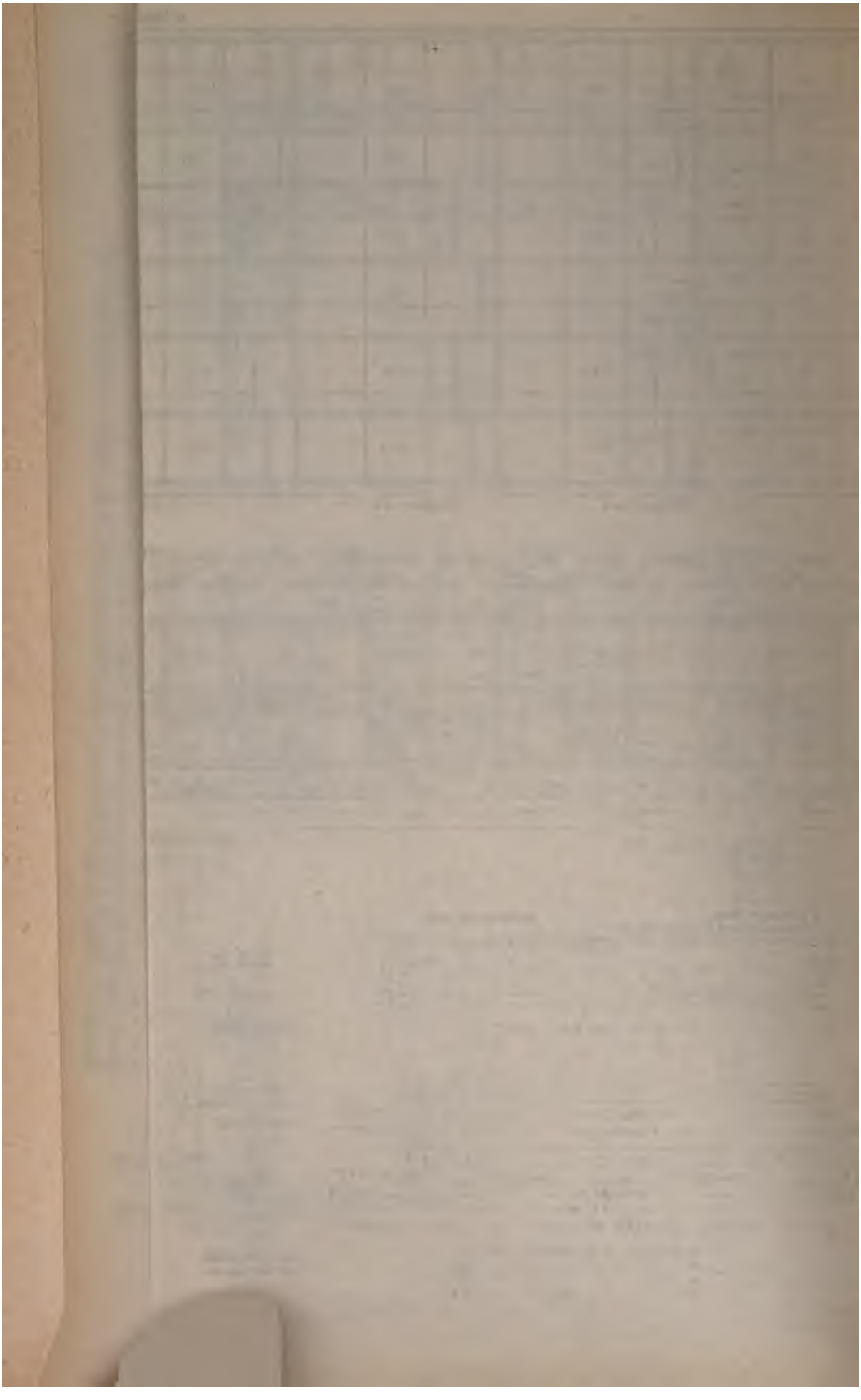




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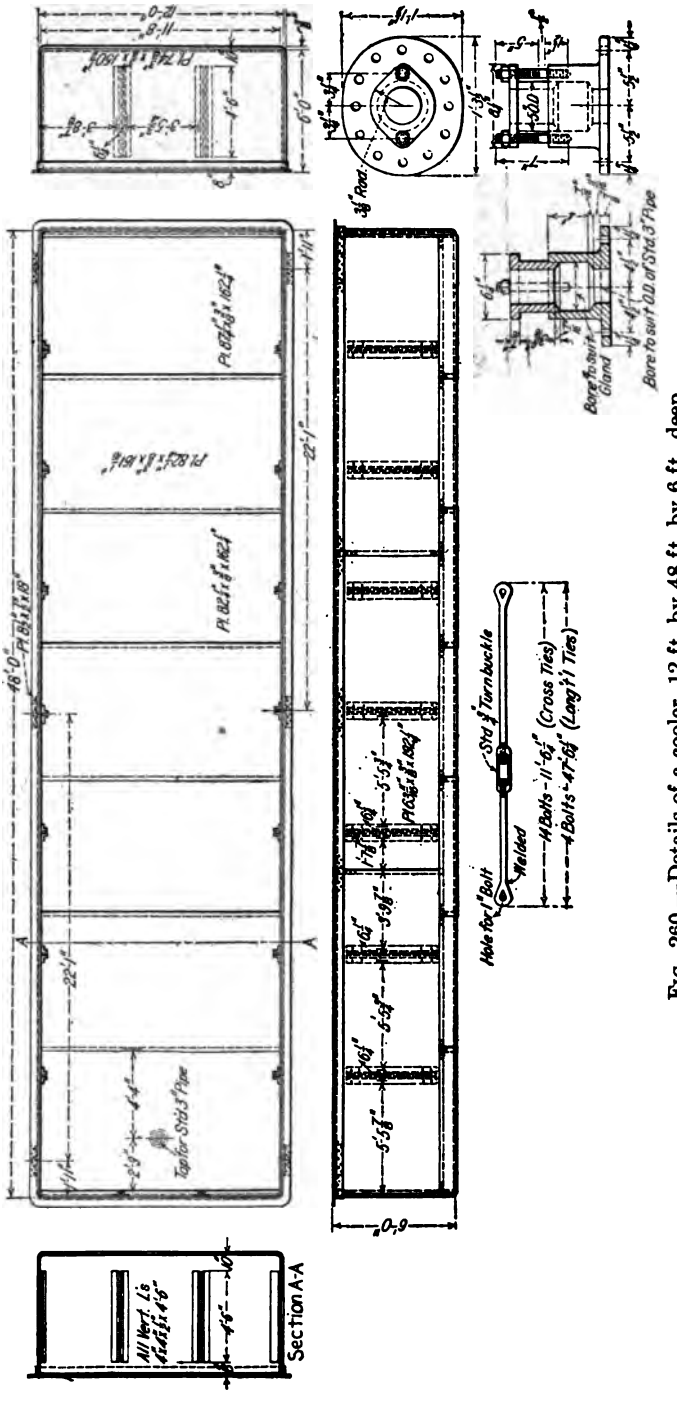



FIG. 260.—Details of a cooler, 12 ft. by 48 ft. by 6 ft. deep.

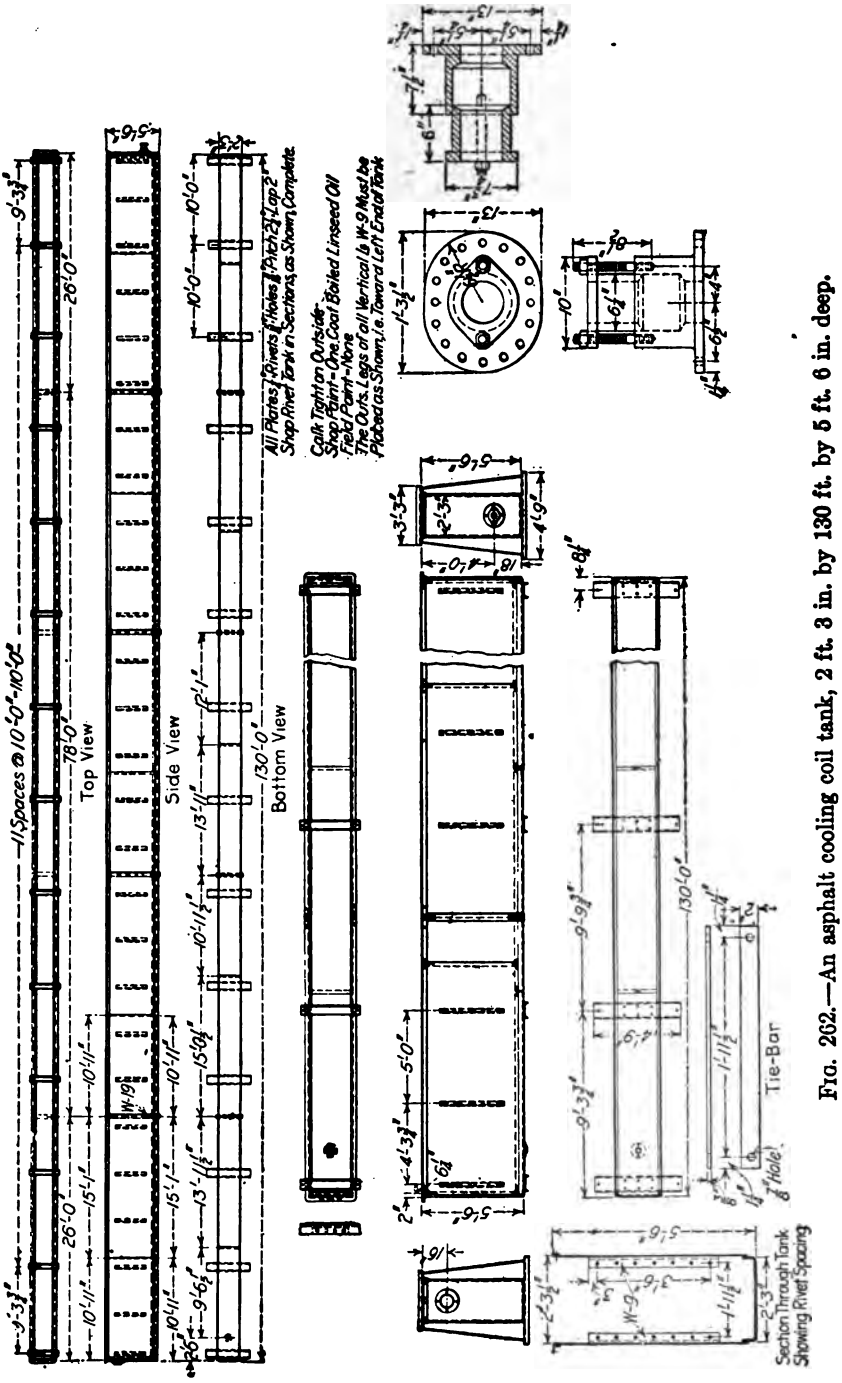



FIG. 262.—An asphalt cooling coil tank, 2 ft. 3 in. by 130 ft. by 5 ft. 6 in. deep.

illustrates a parallel-tube condenser, wherein a number of condensing tubes are carried with a slight fall through a long tank

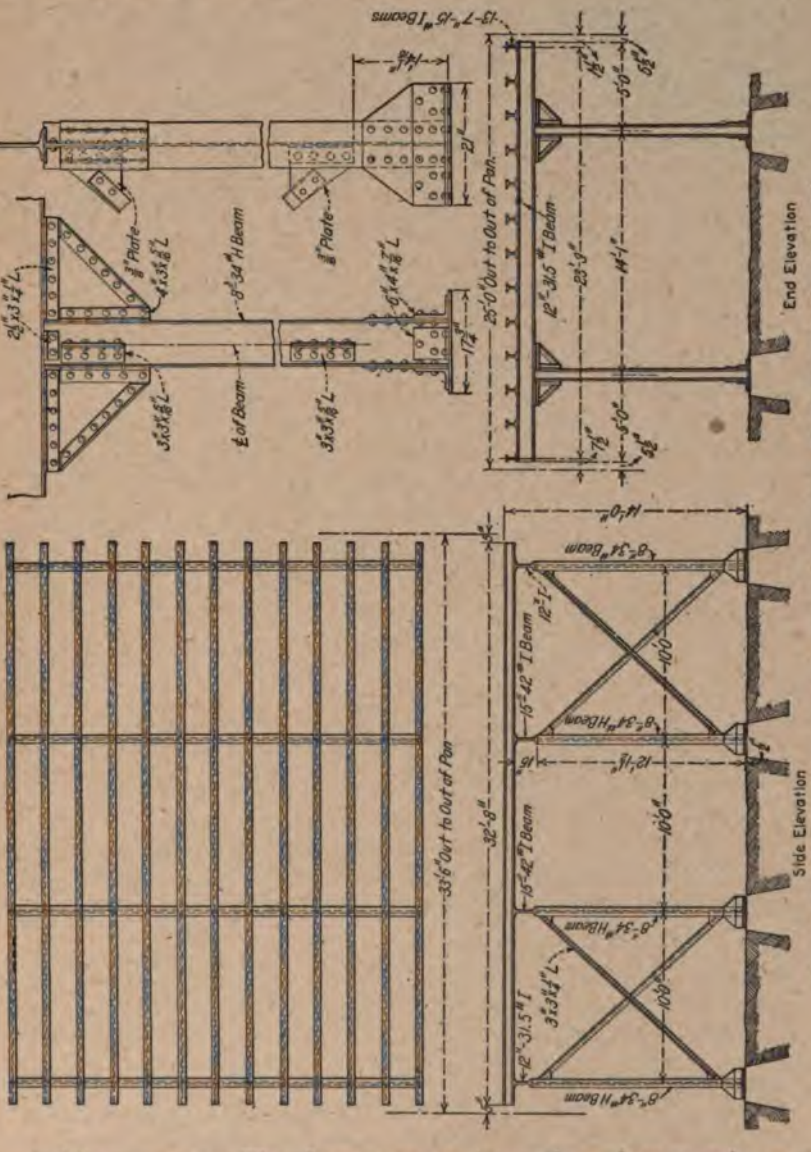



FIG. 263.—Structural support for a condenser box, 25 ft. by 8 ft. by 33 ft. 6 in.

holding water. The Lawrence system of surface condensation consists in the use of a flat box, formed of two sheets of corrugated metal placed close together and joined at the edges;

the vapors circulate between the plates and are condensed by cold water which trickles over the outside.

Figure 257 shows the details of a condenser box, or tank, 23 ft. by 48 ft. by 12 ft. Fig. 258 illustrates three boxes of this design, built for the Huasteca Petroleum Company by the Treadwell Construction Company, of Midland, Pa. Such boxes have a weight of about 57,700 lb. each and are used for stills 14 ft. 6 in. in diameter and 42 ft. in length.

Figure 259 gives the details of the overflow box and diaphragm for a condenser, 30 ft. by 45 ft. by 11 ft. 6 in.; and the design of a cooler, 12 ft. by 48 ft. by 6 ft., is illustrated in Fig. 260.

Figure 261 gives the steel-plate work in the shell for a large condenser box, 105 ft. 4 in. by 29 ft. by 10 ft. high.

In general, it may be said that each refiner has his own opinion regarding the size of condenser boxes and that there is accordingly no fixed rule for arriving at the capacity of the tanks required. However, according to Pennsylvania practice, on 200- to 250-bbl. stills the boxes are usually 30 ft. by 6 ft. by 6 ft.; on 500- to 600-bbl. stills, 40 ft. by 10 ft. by 8 ft.; on 800-bbl. stills, 50 ft. by 10 ft. by 8 ft.; and on 1,000-bbl. stills, 35 ft. by 18 ft. by 8 ft. It is customary for the refiner to furnish and install all necessary condenser tubing.

Typical steel supports for condensers are shown in Fig. 263.

## AGITATORS

The treatment of petroleum products with sulphuric acid and alkali is carried on by agitation with mechanical apparatus or compressed air.

A common practice in refineries has been to perform this agitation by compressed air in vertical cylinders provided with conical bottoms, supported on brickwork, concrete or iron pillars, or a continuation of the agitator shell. When under cover, such agitators may be open above; but if erected in the open air, an iron hood is provided (see Fig. 264). The agitators are usually constructed of wrought-iron plates, and for the acid treatment are lined with lead or "antimonial lead."[1] The thickness of

[1] "Hoyt Metal" sheet is largely used in Pennsylvania refineries for lining agitators. This metal, which contains about 6 per cent. of antimony and is said to have twice the tensile strength of chemical sheet lead of the same thickness, is used in a thickness of $\frac{1}{8}$ in. (8 lb. per square foot) for lining the sides of the agitators and $\frac{5}{32}$ in. (10 lb. per square foot) for the cone.

the sheet lining is usually $\frac{5}{32}$ to $\frac{3}{16}$ in. There are two methods

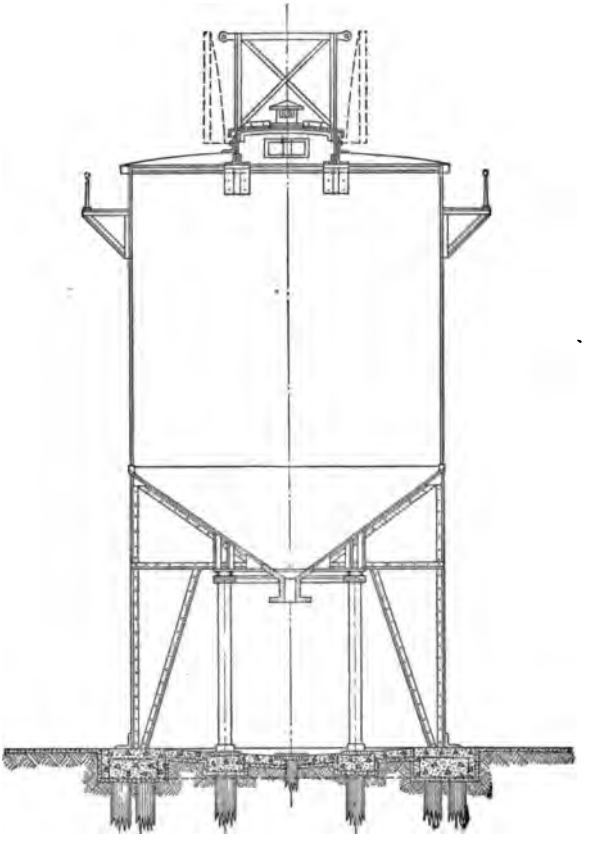


FIG. 264.—One form of agitator.

When sheet lead is used, 10-lb. lead is usually put on the sides and 12-lb. lead on the cone. The following procedure is employed in lining agitators or large round tanks:

Hang one sheet, kink the lower end about every 6 in., then have three or four men pull it back as far as they can and slap it back to the wall two or three times. In doing this, it will nearly fit the sides of the agitator or tank. Continue this until all the side lead is hung, and, when ready to burn, brace each sheet back on either side of the seam (4 in. × 4 in. scantling is used, extending from one side to the other, and moved from sheet to sheet as the seams are burned). If the agitator or tank is 18 or 20 ft. in height, use two braces to each upright sheet (one near the top and the other near the bottom), the upper braces to be handled with ropes, so that they can be raised or lowered to suit. Drive the lead back as the seams are burned up (but do not hammer the seams), then move the braces to the next seam, and so on.

of supplying the air—in one, the usual practice, the air is passed downward through a pipe which extends to the bottom of the

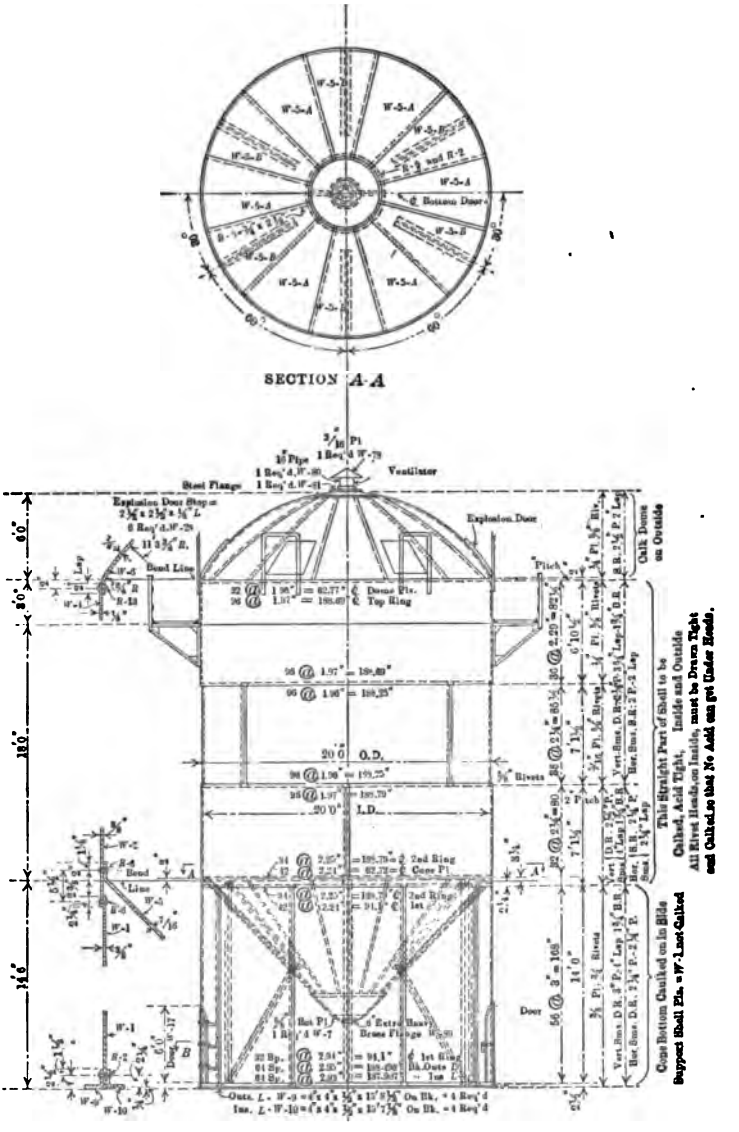



FIG. 265.—A 1200-bbl. agitator, 20 ft. in diameter by 35 ft. 6 in. in height.

cone, and in the other the air passes upward through a pipe opening directly into the bottom of the cone. In most American

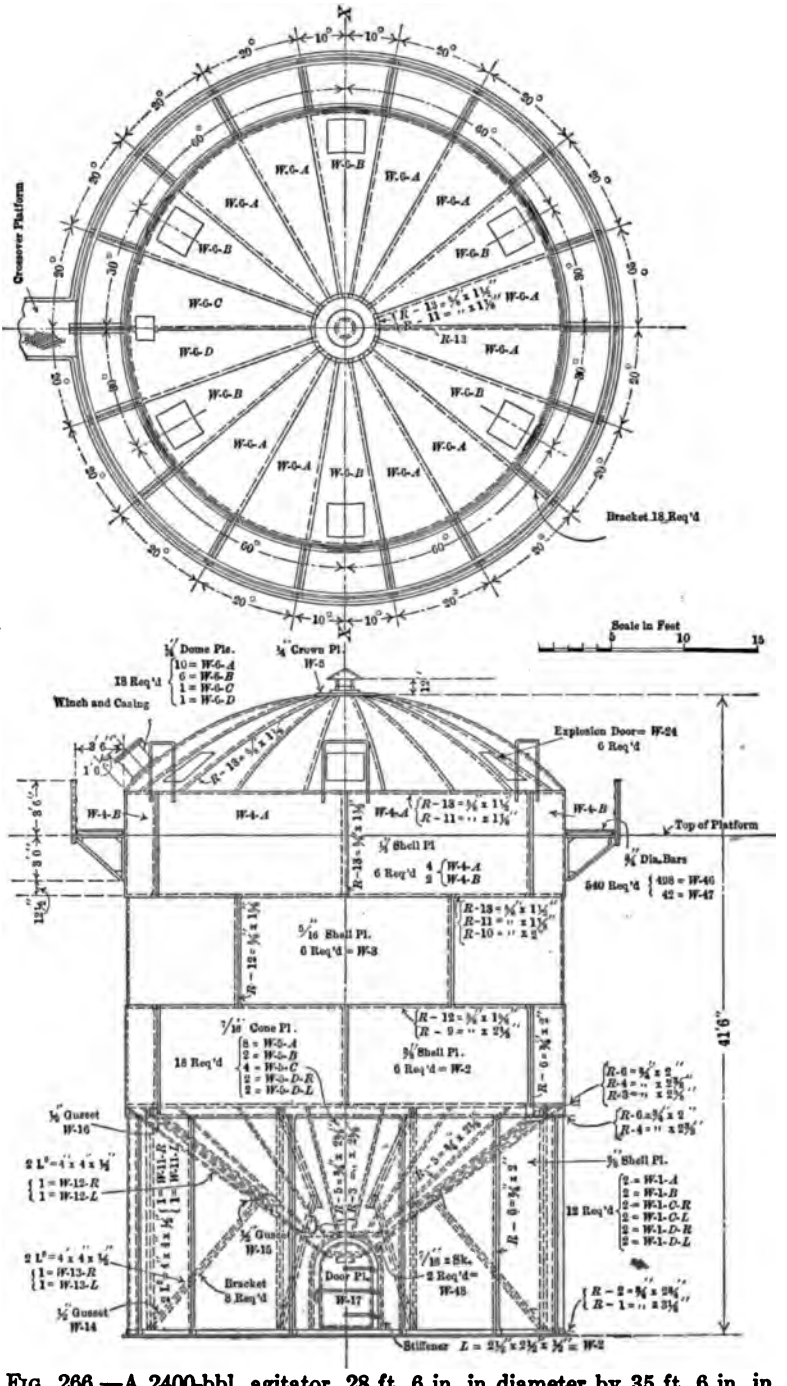



FIG. 266.—A 2400-bbl. agitator, 28 ft. 6 in. in diameter by 35 ft. 6 in. in height.

refineries the acid- and alkali-treatments are conducted in the same agitator; but it has been found, in refineries having a large throughput, that time is saved and a superior product is obtained, when these operations are carried out in separate vessels. The agitator in which the acid-treatment is conducted may be fixed at such a height that its contents will flow into the second agitator for treatment with alkali.

The blowing apparatus may consist of a tube, fitted with a cast-iron rose, shaped like a double cone.[1] A part of the blast

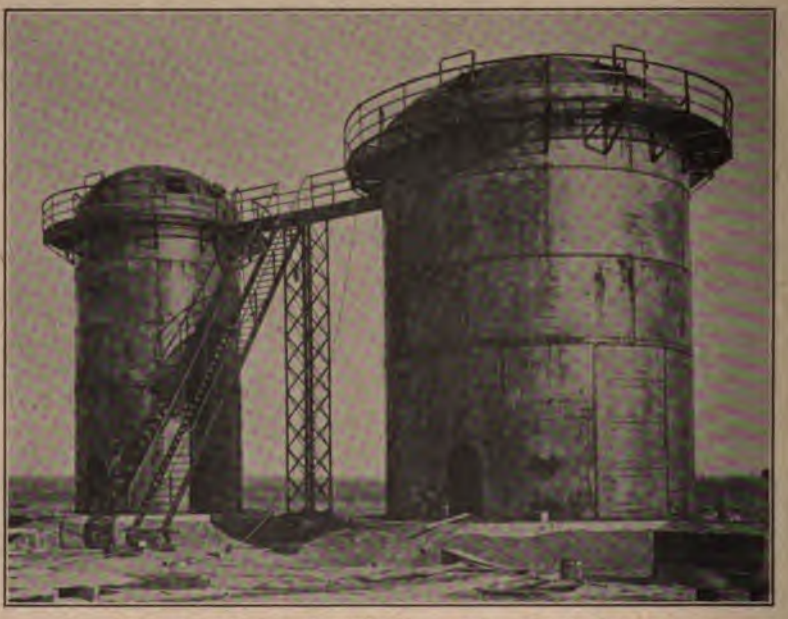
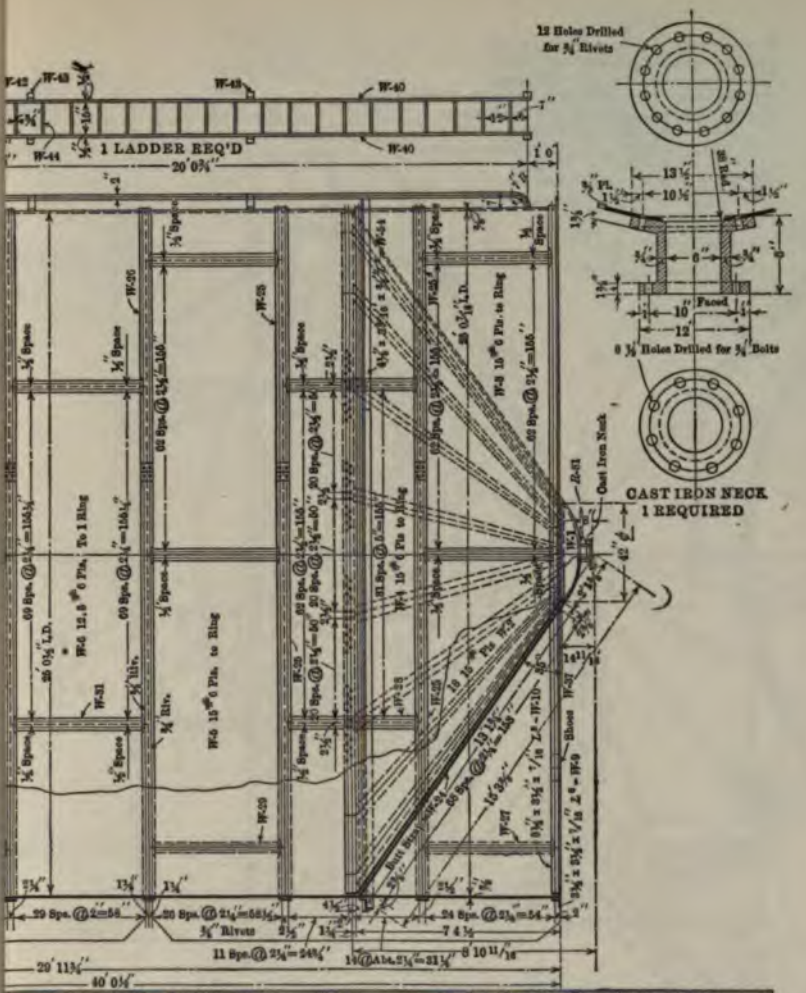


FIG. 267.—1200-bbl. and 2400-bbl. agitators of the latest type.

may thus be directed upon the lowest part of the agitator to stir up the acid sludge there, while the remainder passes into the main body of the liquid and thus sets up a circulation of the liquids. The acid and the alkali for treating the oil are delivered to the agitators through separate pipes by means of a Montejus apparatus. The agitators are usually emptied through three-way branch-pipes with large openings, to allow passage of the thick tarry products. By suitably adjusting the cocks, the

[1] Cf. the agitating apparatus of C. STILL (United States Patent 1080177, Dec. 2, 1913).




| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Agitator | W | 8 | Soft O.H. Steel | 3/16 x 1'-3 | R | 63 | 50 | Rivets Cone Hds. ¾" Dia. x 2½" U.H. | |
| | | 38 | 12 | " " " | x 5' x 15" | | 62 | 1400 | " " " " x 2½" " |
| | | 39 | 15 | " " " | ¾" x 3 x 4' 0" | | 63 | 210 | " " " " x 1½" " |
| Dia. | | 40 | 2 | " " " | ¼ x 2 x 21' 0" | | 64 | 60 | " " " " x 1⅝" " |
| 5/16 | | 41 | 2 | " " " | " x " x 14' 8" | | 65 | 170 | " " " " x 1½" " |
| x 157¼" | | 42 | 2 | " " " | " x " x 10' | | 66 | 210 | " " " " x 1¼" " |
| " x " | | 45 | 6 | " " " | " x 3' x 11" | | 67 | 205 | " " " ¾" " x 2½" " |
| x " | | 44 | 33 | " " " | ¾" Dia. x 1" 4½" | | 68 | 20 | " " " " " x 2¼" " |
| " x " | | 45 | 16 | Bolts Hex. Hd.& Nuts ¾" | " x 5" U.H. | | 69 | 80 | " " " " " x 2" " |
| 5/16 | | 46 | 8 | Soft O.H. Steel | 10"2 x 34" x 40" | | 70 | 40 | " " " " " x 1¾" " |
| " | | 47 | 1 | | 15" x 10" x 23¼" 4'9"05 | | 71 | 80 | " " " " " x 1¾" " |
| x 9'8 x 20' 0" | | 48 | 1 | Std. Cast Iron | 6" Covered Flange | | 72 | 220 | " " " " " x 1¾" " |
| x " x 12' 0" | | 49 | 216 | Bolts Hex. Hd.& Nuts | ¾" Dia. x 1½" U.H. | | 73 | 980 | " " " " " x 1½" " |
| x 6'1 x 12'1" | | 50 | 1 | Flanges Tapped for 2" Gas Pipe | | | 74 | 2980 | " " " " " x 1⅝" " |
| x " x 4'7" | | 51 | 1 | 2" Gas Pipe Nipples 10" Lg. | | | 75 | 200 | " " " " " x 1¼" " |
| x 4'1 x 19'11" | | 52 | 2 | 2" Elbows | | | 76 | 20 | " Shop Rivets " x " " |
| x " x 12'3" | | 53 | 1 | 2" Gas Pipe Nipples 2" Lg. | | | 77 | 50 | " " " " " x 1¾" " |
| x " x 6'6" | | 54 | 4 | 2 Bars, O.H. Steel | 4½" x 3/16 x 12'4 x 20' 1 | | 78 | 160 | " " " " " x 1⅝" " |
| x " x 8'3" | | 55 | 4 | Bars " " | ¾" x 6⅝" x 10" | | 79 | 80 | " " " " " x 1⅝" " |
| x " x 8'2" | | 56 | 8 | " " " | ¾" x 5¼" x 2'6" | | 80 | 90 | " " " " " x 1¼" " |
| x 9'8 x 0'6" | | 57 | 8 | " " " | ¾" Dia. x 1'3" Lg. | | | | |
| x 4'1 x 11'6" | | | | Field Rivets | | | 81 | 1 | Cast Iron Neck |
| x 9'8 x 0'5¼" | | R | 58 | 60 | Rivets Cone Hds ¾" Dia. x 2½" U.H. | | R | 81 | 12 | Rivets Cone Hds. ¾" Dia. x 2" U.H. ( Field ) |
| 12'x 66" | | | 59 | 80 | " " " " " x 2¼" " | | | | | |
| 5¼" Dia. | | | 60 | 420 | " " " " " x 2½" " | | | | | |
| x 14½" U.H. | | | | | | | | | | |
| x 13'1½" | | | | | | | | | | |
| x 12'1" | | | | | | | | | | |
| x 15'1" | | | | | | | | | | |
| x 4'2⅜" | | | | | | | | | | |
| x 4'8" | | | | | | | | | | |
| x 4'9½" | | | | | | | | | | |
| x 18'6⅜" | | | | | | | | | | |
| x 4'2⅜" | | | | | | | | | | |
| x 1'0" | | | | | | | | | | |
| x 0'10" | | | | | | | | | | |
| x 0'10" | | | | | | | | | | |
| x 1'0" | | | | | | | | | | |
| x 12'7" | | | | | | | | | | |

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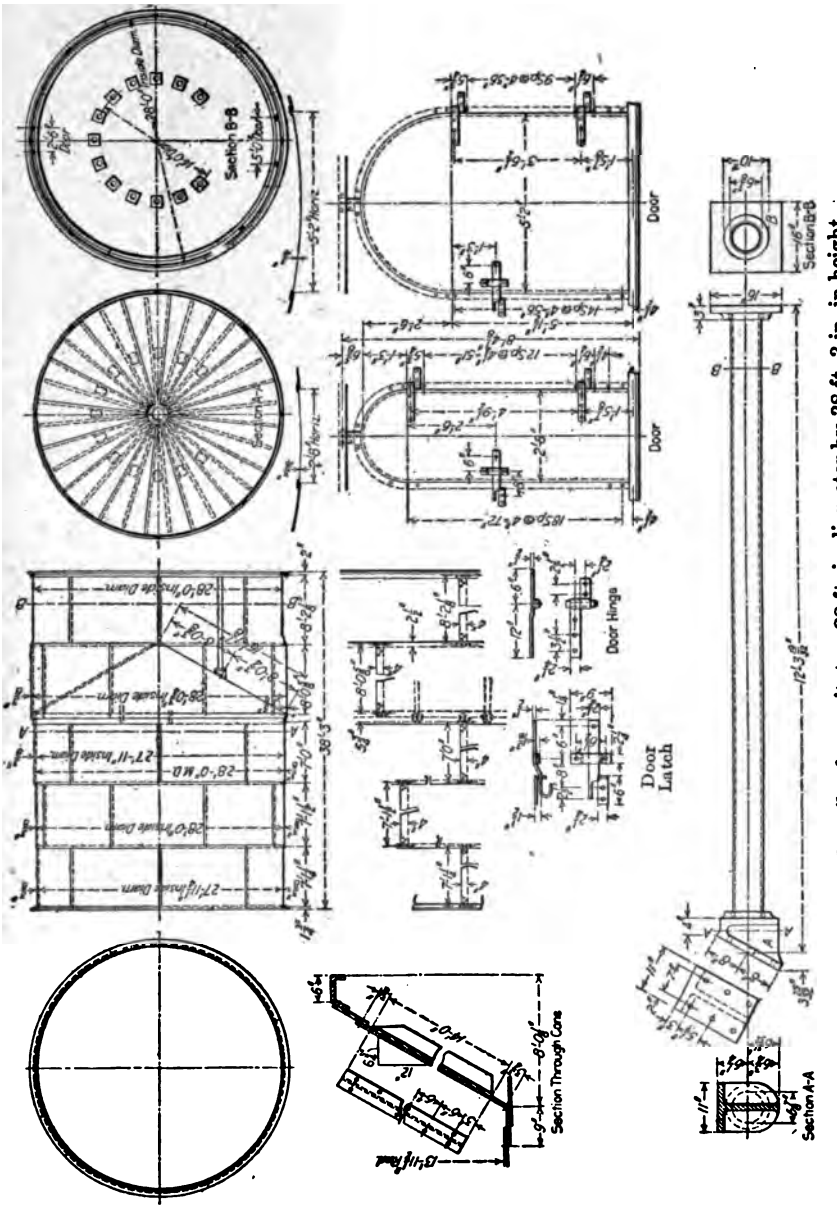



FIG. 269.—Details of an agitator, 28 ft. in diameter by 38 ft. 3 in. in height.



FIG. 270.—The two top rings and globe roof for an agitator.



FIG. 271.—Two agitators provided with a common stairway.

waste acid, or alkali, and the oil, are conveyed to their proper receptacles.

For reasons which have been indicated, some refiners, especially certain producers of specialties, prefer to use mechanical

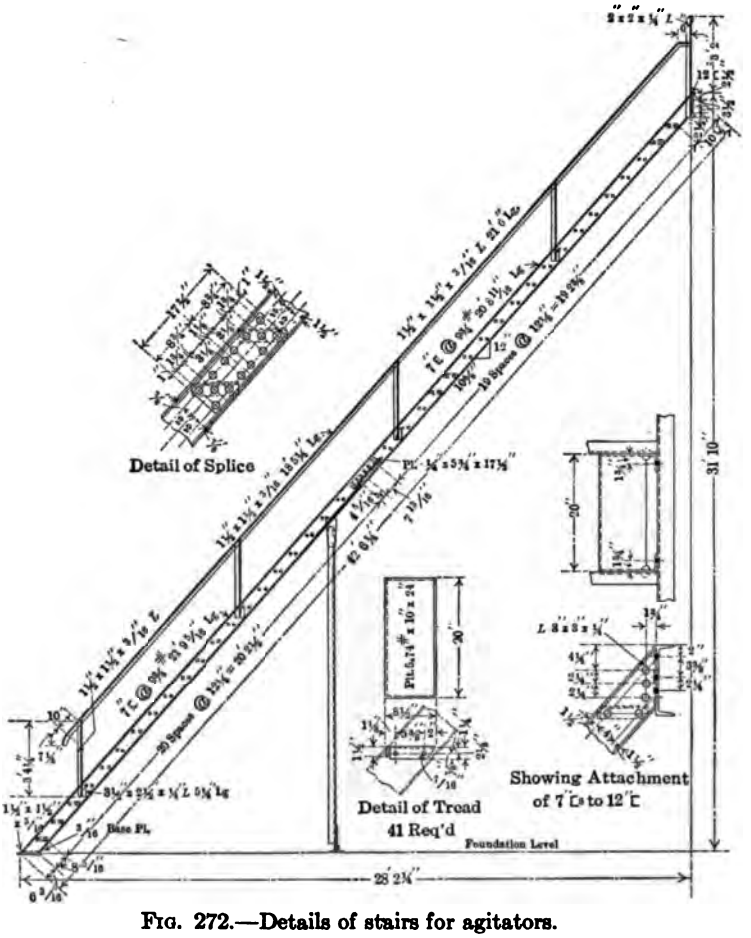



FIG. 272.—Details of stairs for agitators.

agitators in the chemical treatment of lubricating oils. Such agitators have also been employed in the refining of benzine, but at present the air-blast system is most in favor and is in general use for the treatment of *all* distillates. It has been maintained by some engineers that the agitation of light distillates by air

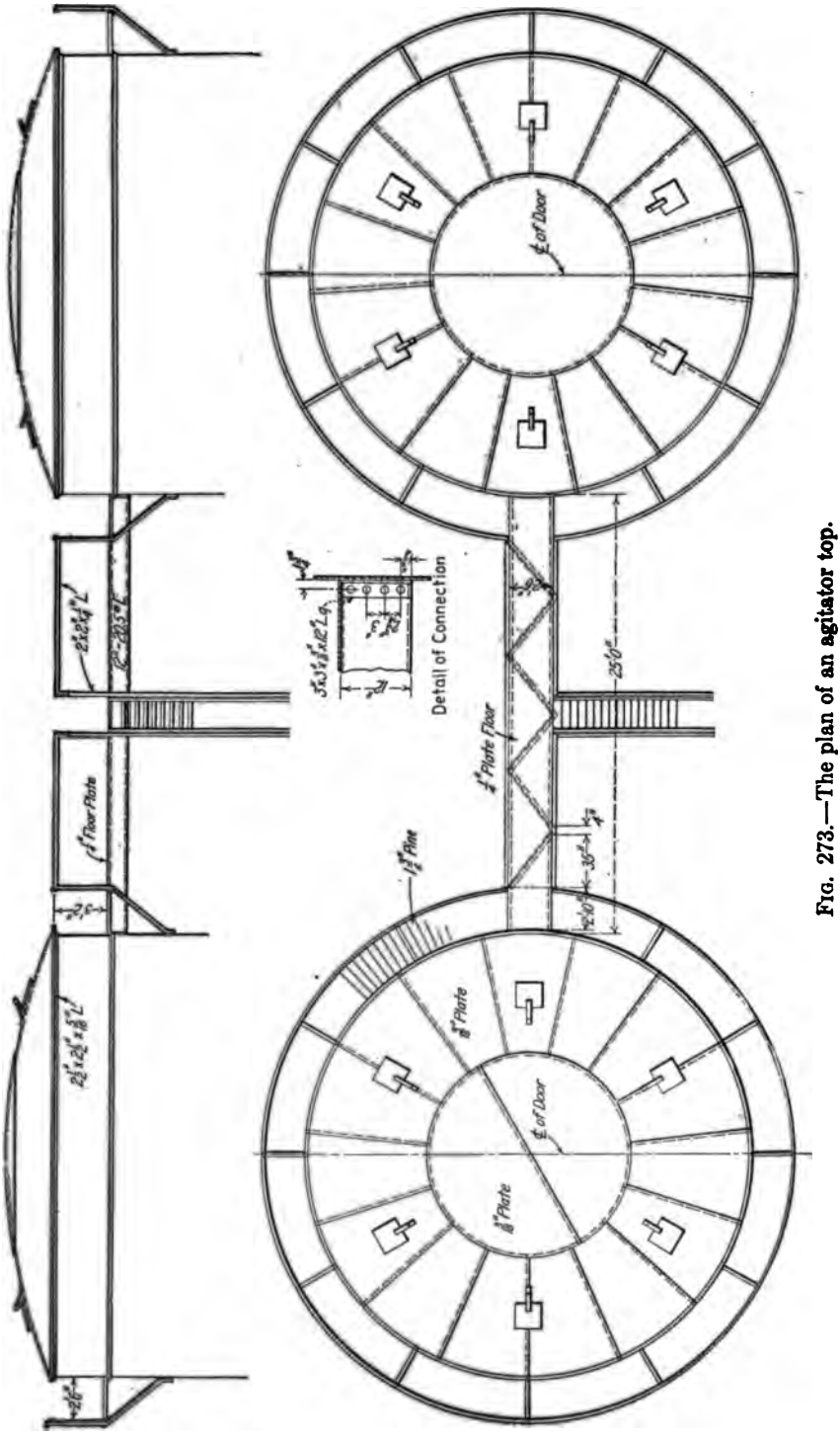
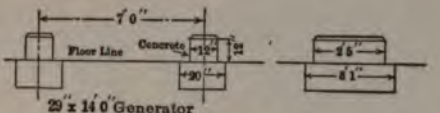
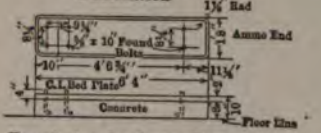


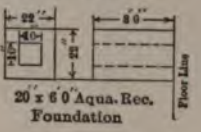

FIG. 273.—The plan of an agitator top.



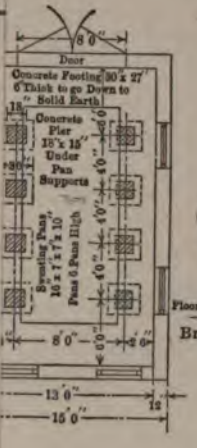

29" x 14' 0" Generator Foundation

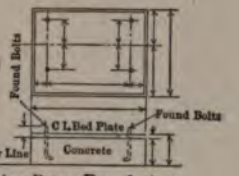



Foundation for Ammo Pump 8" x 3¼" x 12" C.M.Co.

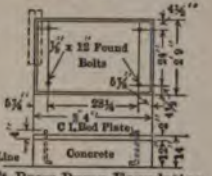



20" x 6' 0" Aqua. Rec. Foundation

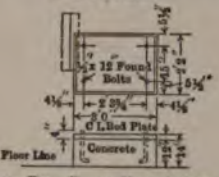



Brine Pump Foundation 5 x 8 Goulds




Soft Press Pump Foundation 2¼" x 6 Goulds

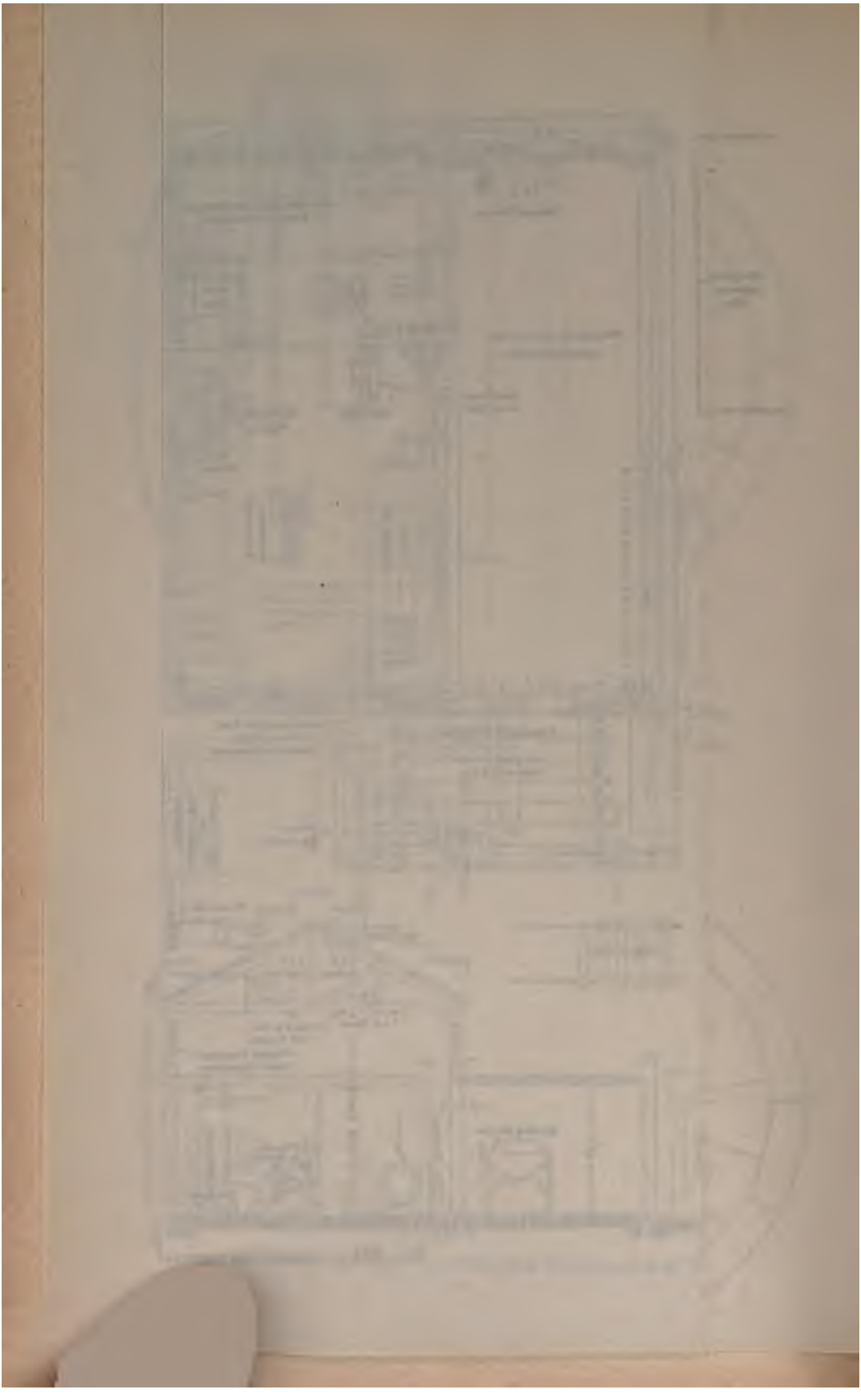



Ram Pump Foundation 1¼" x 6 Goulds




Warren, Pa.

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is out of the question unless a closed agitator is employed and the whole of the air supply to the air pump or "blower" is drawn from the top of the tank, in which case any vapors passing off in the air are returned in the air delivered from the blower; but it has been found in practice that, with the deep form of agitator now in use, crude benzine, when cold (direct from the condensers), may be agitated with sulphuric acid by air, in an open agitator, with a maximum loss of 2 per cent. by volume.[1]

Mention may be made here of chemical treatment of gasoline and naphtha without the use of agitators. In one arrangement in which agitators are dispensed with, the light distillate flows successively through vessels respectively containing sulphuric acid, caustic soda, washing water, and finally a filtering mixture of sawdust and salt. The first three vessels contain the sulphuric acid. The vessels, which are all closed, are about half-filled with the reagent, and are arranged at successively lower levels. The spirit is run into the first through a perforated tube passing to the bottom of the vessel, rises through the reagent, and finally escapes through a tube at the top of the vessel into the next. It is thus successively acted upon by acid, alkali, and water, and passes through the filter into a receiver.

Figure 265 shows a 1,200-bbl. agitator, 20 ft. in diameter and 35 ft. 6 in. in height. A 2,400-bbl. agitator, 28 ft. 6 in. in diameter and 35 ft. 6 in. in height, is detailed in Fig. 266. Fig. 267 shows agitators of these sizes, recently erected for an American refining company.

The detailed construction of an agitator of the general type now in use is presented in Fig. 268. The two top rings and the globe roof for an agitator are exhibited in Fig. 270. Fig. 271 shows two agitators erected in the field, with a common stairway. Most refiners now favor a low globe roof, fitted with explosion doors.

## PARAFFIN WAX PLANT EQUIPMENT[2]

The general arrangement and foundation plan of a paraffin wax plant in the Appalachian field, having a capacity of 90,000 lb. (400 bbl.) per month, is given in Fig. 274. The absorption

[1] Pennsylvania refiners regard the losses by air agitation as satisfactory when this operation is carefully conducted.

[2] For a general descriptive account of the manufacture of paraffin wax, see p. 478 *et seq.*

machine[1] of 60 tons refrigerating capacity furnishes brine to the wax chilling and pressing plant at a temperature of −15°F. to −20°F. In this plant the requirements are for 0°F. cold-test neutral oil, and it is therefore necessary to maintain this low temperature and to press twice, an operation known as "double-pressing" and requiring additional chilling machines and filter presses. In one operation, or "single-pressing," finished neutral oils are made which show a cold test of 15° to 25°F. and less apparatus is required for a given quantity of unpressed distillate.

This plant has three 12-section distillate chilling machines and five 48-in. filter presses. Four of these presses, aggregating 800 filter-press plates, are used on the first pressing, and the largest press, containing 450 plates, is used on the second pressing.[2]

The following description is furnished by the builder, the Carbondale Machine Company, of Carbondale, Pa., of a modern wax plant installed in the early part of 1915 for the Gulf Refining Company at Port Arthur, Tex. (see Fig. 275). The daily capacity of this plant is 1,500 42-gal. bbl.

The wax distillate is stored in tanks of suitable size and pumped into the wax plant for the purpose of separating the wax from the oil. It is necessary to chill and granulate this distillate, and for this purpose two 100-ton absorption refrigerating machines are used. These are of the atmospheric type, having coils arranged on the roof with distributing troughs, so that water passes by gravity on the outside of the pipes of the condenser, absorber, weak liquor cooler and rectifier. Steam ammonia pumps having automatic regulators are used. There is an extra ammonia pump, which is used as a reserve for either machine. The brine is pumped through a shell cooler and thence through the four horizontal double-pipe chilling machines. This cools the unpressed wax distillate to within 10° or 12°F. of the temperature of the inlet brine. The distillate is in the inner pipe and, when chilled, becomes solidified. This inner pipe has a slow revolving helicoid conveyor, which scrapes the walls of the pipe, having a tendency to granulate the distillate, which af-

[1] The absorption refrigerating system has been found to be best adapted for the manufacture of paraffin wax and neutral oils (see p. 756).

[2] The wax obtained from the first pressing is either marketed as high melting-point wax or is mixed in suitable proportions with the wax obtained from the second pressing to make wax of such lower melting point as is required.

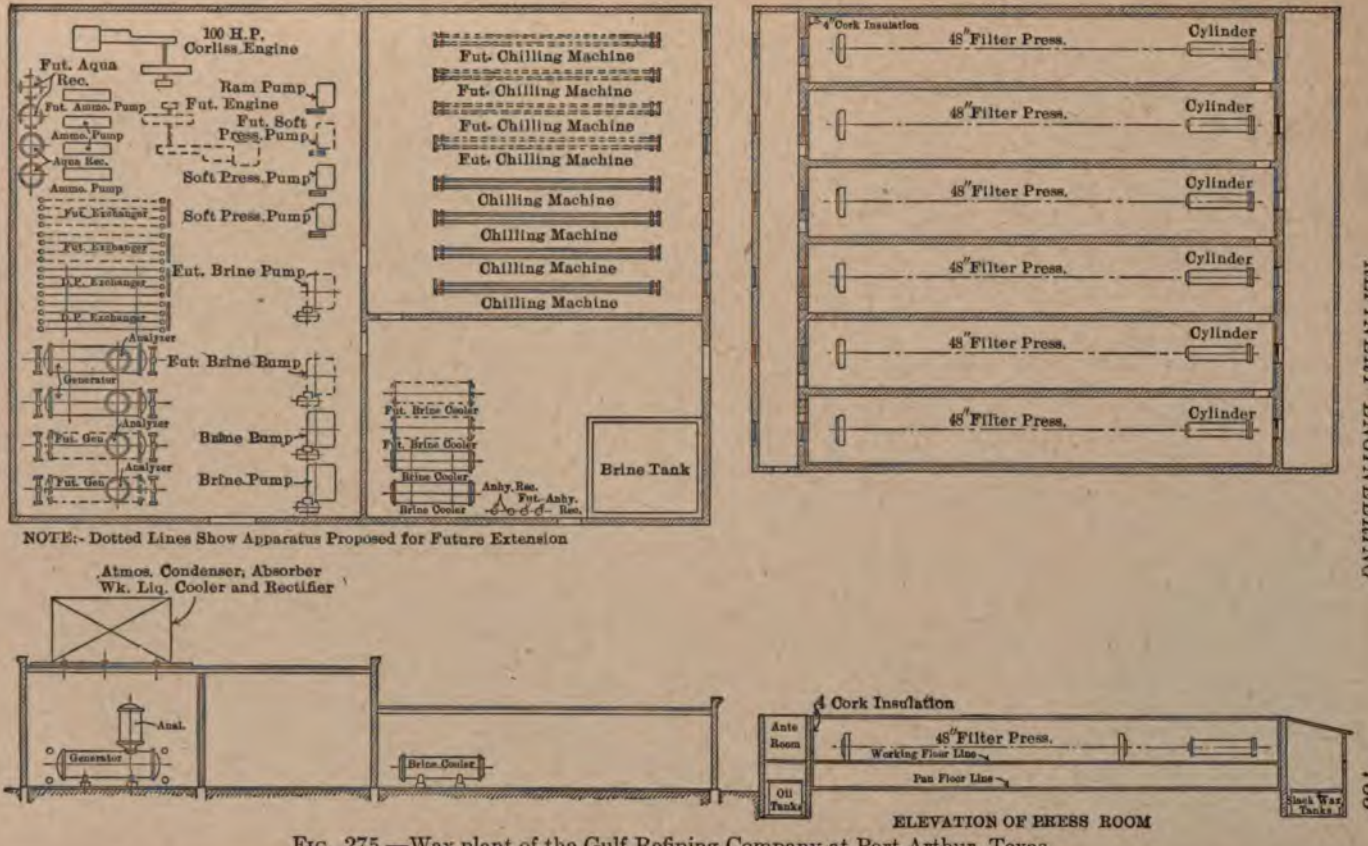



FIG. 275.—Wax plant of the Gulf Refining Company at Port Arthur, Texas.

fords a good separation in the filter presses. The cold brine is circulated in the annular space of the chillers and returned to a brine storage tank. The wax distillate, after being chilled, is delivered to the presses.

For separating the wax from the pressed oil, six high-pressure, hydraulic-ended filter presses are used. Each press has 500 48-in. plates with ½-in. spacing rings, and is provided with double troughs and conveyor. One trough is to catch the pressed oil; and after the press has finished dripping, it is removed to one side on rollers by means of a shifting lever. The press is then ready for dumping, as the "slack wax" has accumulated in the spacing rings, the pressed oil having passed through heavy cotton blankets. The filter press piston ("ram") is set back so as to allow the press men to remove the cakes by means of spuds. The "slack wax" falls to the lower trough and is removed to tanks by means of a helicoid conveyor. It is then melted and pumped into sweating pans for removing more oil; and, when finished and filtered, is run into moulding machines and turned out as refined wax containing less than 1 per cent. of oil and moisture.

The pumping equipment consists of two soft press pumps for delivering the distillate to the chillers, thence to the presses; one setting-up pump; two brine pumps; two pressed-oil pumps; one melted slack wax pump; one melted sweated wax pump; and one foots oil pump, all of which are belt-driven power pumps.

**Distillate Chilling Machine.**—In practice, the "wax distillate" is first cooled, usually to a temperature of 15° to 20°F., depending upon the cold test of the pressed oil required, to produce which chilling effect an ammonia absorption type of refrigerating machine is used.[1]

One type of chiller consists of a series of double pipes with heavy cast-iron fittings; and the inner or 6-in. pipe has a slowly revolving helicoid conveyor, which scrapes the walls of the pipe. Fig. 277 shows the general arrangement of a machine with the chain and driving sprockets. The speed of the conveyor is about 8 r.p.m. The chain is driven by means of reduction gears, either of the worm-gear or bevel-gear type.

**Filter Presses.**—Fig. 278 shows a Carbondale filter press, 48 in. in diameter, provided with a drag conveyor and holding 300

[1] See account on p. 479.

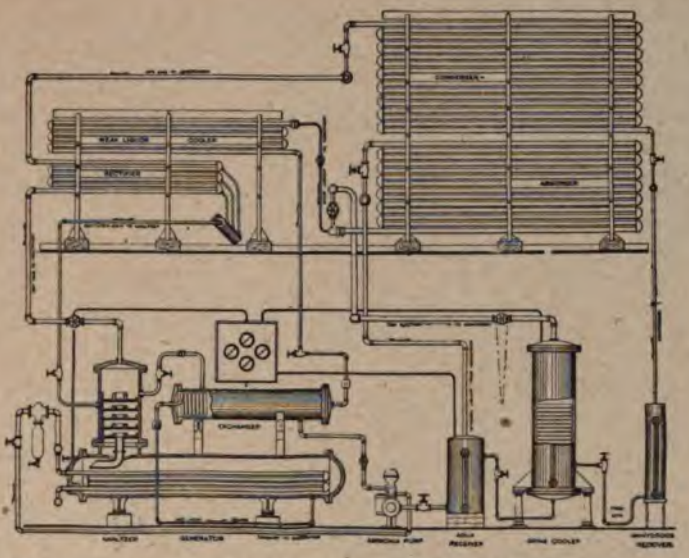


FIG. 276.—Cross-section of an atmospheric refrigerating machine.

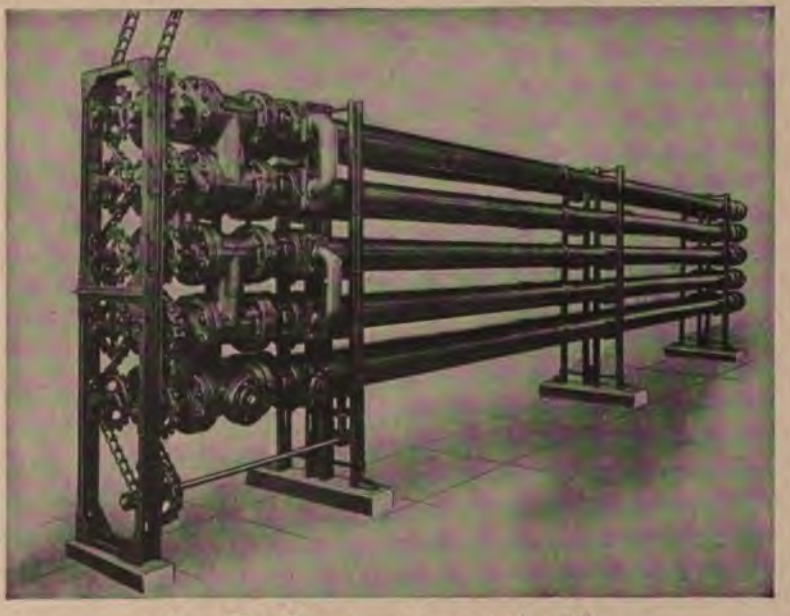


FIG. 277.—General arrangement of a chiller.

plates and 299 ½-in. rings. Fig. 279 presents an interior view of the soft press room of a Kansas refinery containing three 500-plate Moore presses. A 30-in. square cast-iron Moore press is shown in Fig. 280.

Figure 281 illustrates a 48-in. soft press manufactured by the Carbondale Machine Company. Presses of this type are in use at a number of Pennsylvania refineries.

The general arrangement of a 48-in. soft press and a chilling machine is given in Fig. 283.

*Experimental Presses.*—Many of the large refineries are provided with a small room, well insulated and cooled by a series

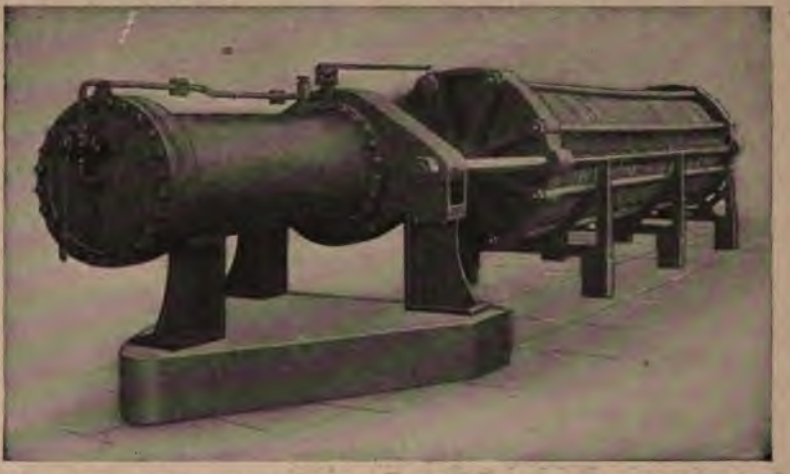


FIG. 278.—A Carbondale filter press, 48 in. in diameter, provided with a drag conveyor.

of brine coils, wherein are installed small presses for experimental purposes.[1] The results obtained from such experiments not only indicate the method of handling the distillate in large quantities, but also serve as a check on the yields obtained in a large plant.

A Moore experimental press, 12 in. in diameter, is shown in Fig. 284. This press contains 12 filter plates and eleven ½-in.

[1] The following are some of the usual pressing plant troubles which may be solved by systematic experimentation: An occasional batch of wax distillate (see p. 475) which is difficult to press; failure to obtain the whole of the recoverable wax from the distillate; difficulty in securing a cold test on the finished oil, and differences in results from pressing a stock "green" (just as it leaves the condenser) and after preliminary treatment.

rings; it is 5 ft. 6 in. in length over all and weighs 1,600 lb. The supports are bolted to a cast-iron bed plate, which also acts



FIG. 279.—An interior view of a soft press room containing three 500-plate Moore presses.

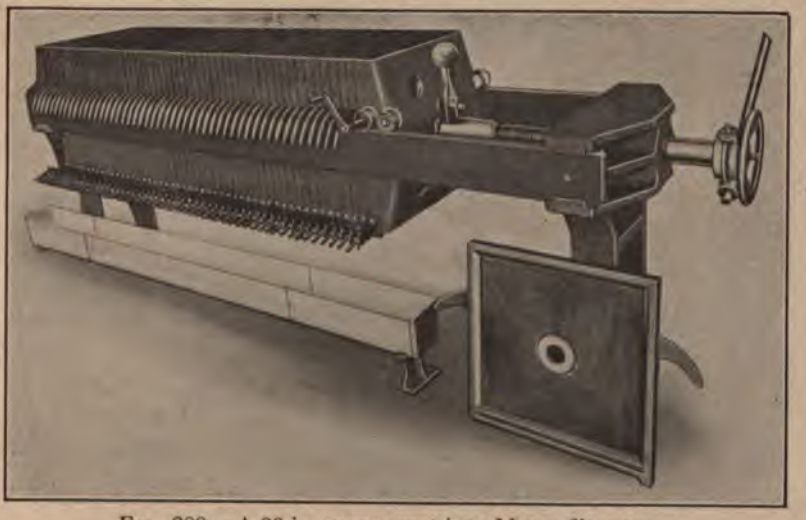


FIG. 280.—A 30-in. square cast-iron Moore filter press.

as a pan for receiving the pressed oil. To this same bed plate is attached a hydraulic hand-pump, making the machine a

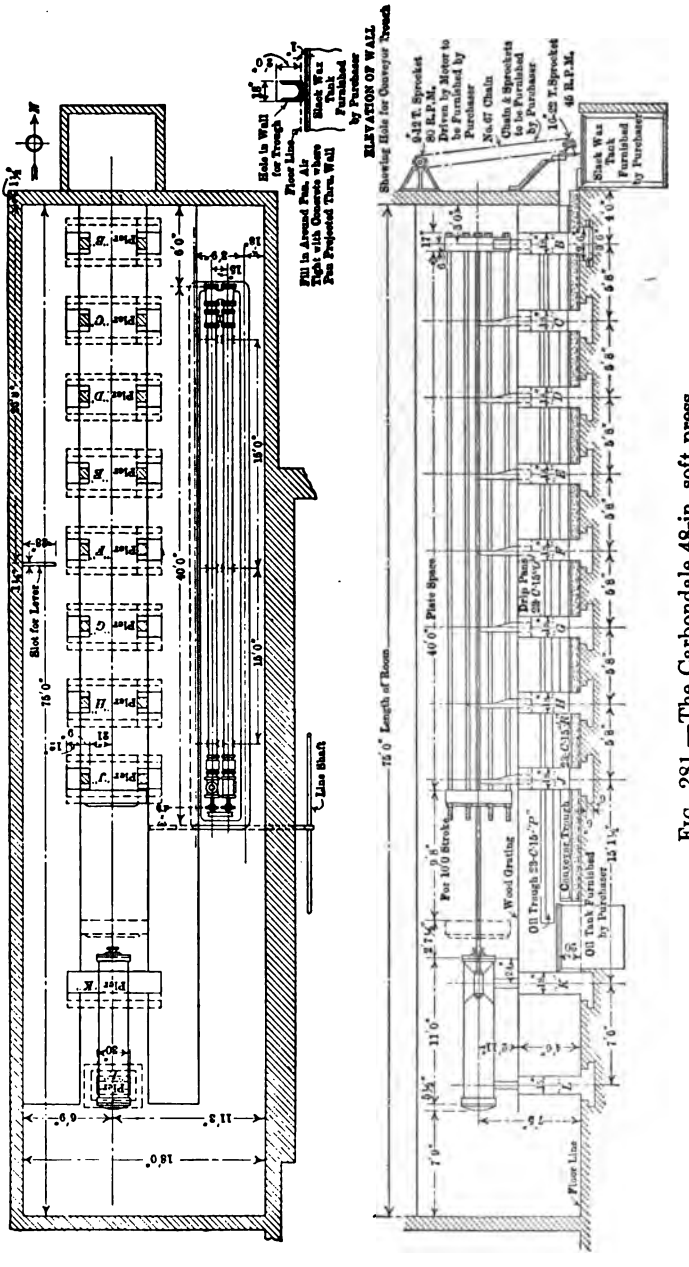



FIG. 281.—The Carbondale 48-in. soft press.

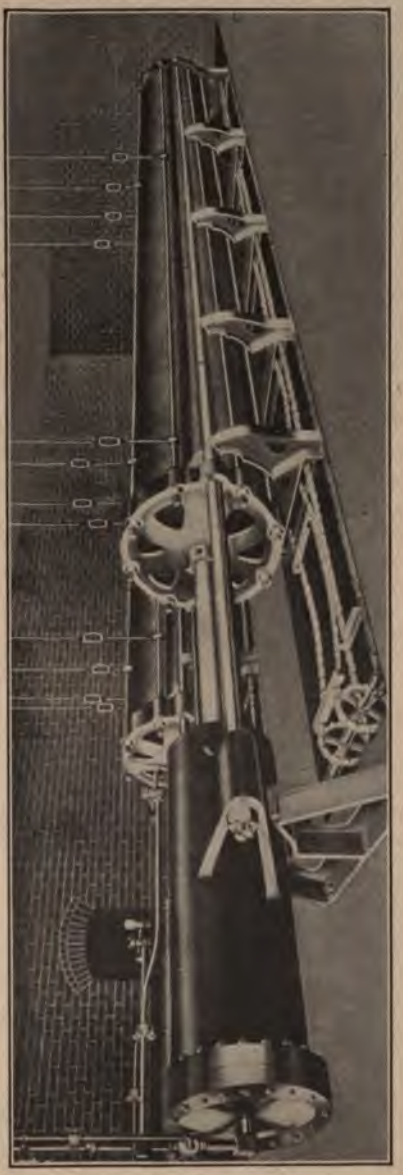


FIG. 282.—Moore 48-in. soft presses.

complete pressing plant on a small scale. It is built to stand 400-lb. pressure, so that one could, if necessary, connect it on

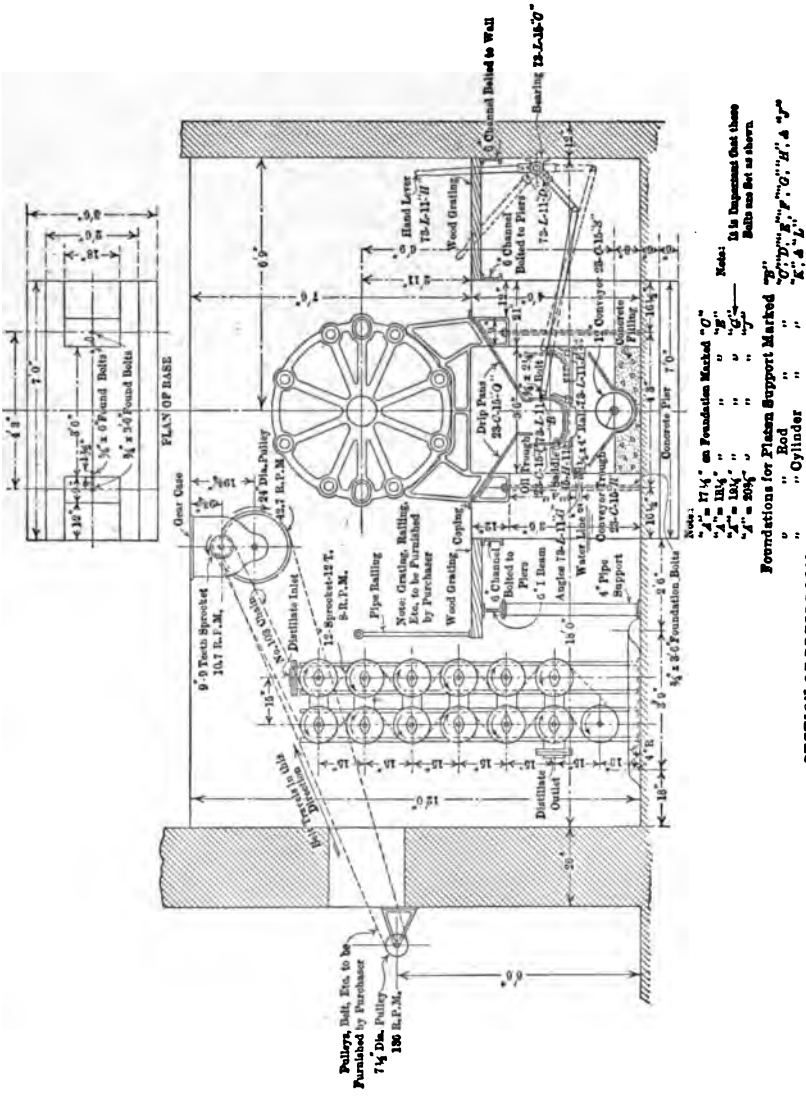



FIG. 283.—General arrangement of a 48-in. soft press and chilling machine.

the main discharge line to larger presses, securing thereby an indication of what a distillate should yield without having to wait 24 hr. for a big press to dry out.

**Sweating Pans.**—The elevation of a modern sweating outfit ("scale wax sweater") is presented in Fig. 285.

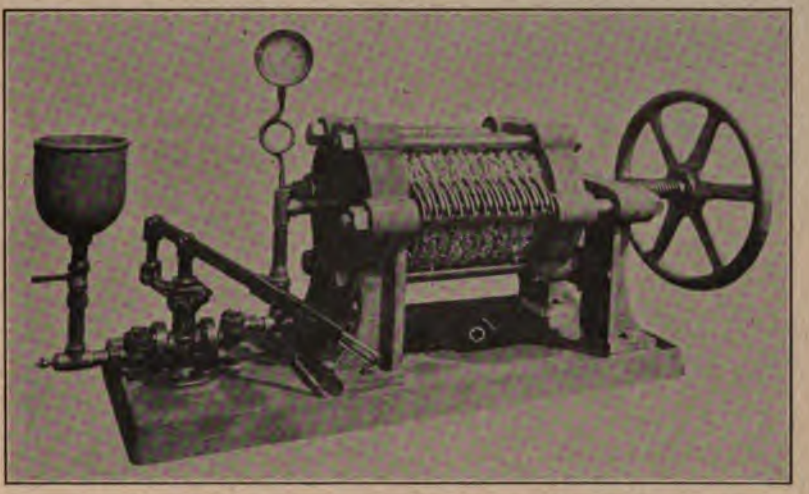
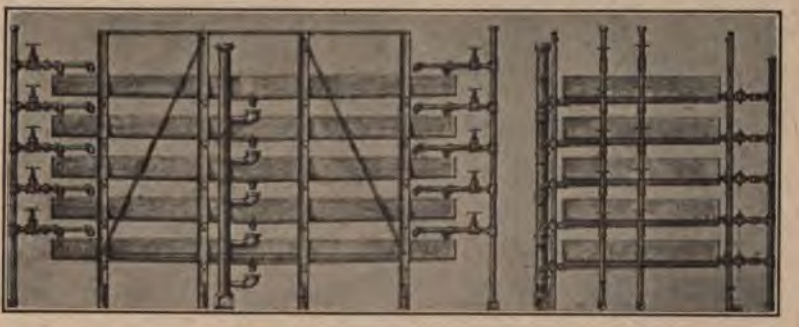


FIG. 284.—A filter press for experimental purposes.

**The Sweating Process: Engineering Considerations.**—Very little information has been published regarding the important sweating process and not infrequently the statements of refiners are of a confusing nature. The variability of petroleum and



Side view. End view.

FIG. 285.—The elevation of a modern sweating outfit ("scale wax sweater").

variations in the methods of distillation and pressing are largely responsible for this condition. However, the actual sweating of paraffin wax is practically the same in all cases,[1] the only

[1] For a brief general description of this process, see p. 479.

difference in operation being one of temperature, both as to circulating water and the oven.

*The Building.*—The building in which sweaters are located, or, as it is sometimes called, the "oven," is a necessary feature of a sweating system, both as to location and construction. It should be a substantial building, either of brick or concrete, and should be so located that there will be free circulation of air on all sides. All doors and windows should be large, easily opened, and so constructed that, when closed, they will make the oven practically air-tight. This last feature is especially important, since any air leakage affects the evenness of the room temperature and makes steam cost unnecessarily high.

*Pans.*—There are many different sizes and shapes of pans in use at the present time, all of which seem to be giving satisfactory results; but the Samuel L. Moore and Sons Corporation considers 8 ft. wide by 20 ft. by 14 in. deep, each stack to have 8 pans, as the best size for small plants. Pan stacks should be so designed that the entire inside surface will drain to a common point, usually at the center of the 20-ft. side, and this pitch of the pans must be accurate, in order to prevent imperfect drainage and, as a result, an inferior quality of wax. About 6 in. below the top of the pan, and covering the entire inside area thereof, there should be a galvanized-iron or brass-wire screen of mesh. This screen should be securely fastened to angle irons which are riveted to the sides of the pan. Care should be taken in fastening this screen, for it must be very taut and perfectly level.

*Melting-down Coil.*—This coil is not absolutely necessary and some refiners consider it entirely useless, but it has been found that the installation of such a coil enables one to empty a pan in much less time than is required where pans are not so equipped. The coil is usually made up of a main line with several branches. The branch pipe has a number of small holes in one wall, located at regular intervals, and in a position suited to melt the wax rapidly.

*Water-circulating System.*—This system is one of the indispensable features of sweater plant design, although it frequently receives but little attention. The coil should be of the correct size and location, and one should have as perfect control of the water temperature as is possible. The work performed by the coil itself is most important, as this feature alone practically determines the capacity of sweaters. The functions of this coil

are: first, to chill the molten wax preparatory to letting it down on the screen, which is accomplished by circulating cold water; and, second, to warm the entire charge, gradually and evenly, until it reaches the sweating temperature, this being performed by the hot water system. Inasmuch as the time consumed in emptying, charging, cooling and warming to the required temperature must be added to the actual time required to sweat a charge of wax, it is apparent how important it is to have this accomplished as rapidly as possible and to what extent this coil affects the sweater capacity.

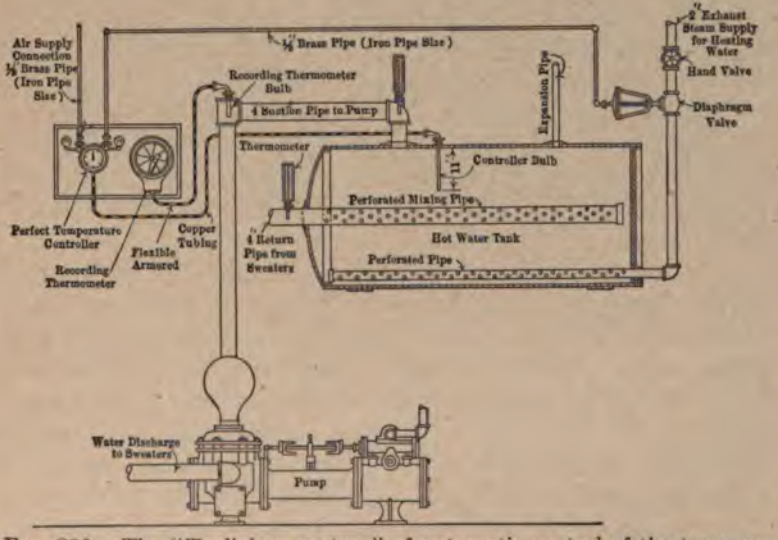



FIG. 286.—The "Tagliabue system" of automatic control of the temperature of paraffin wax sweaters.

The hot water system is essential only so far as control of the temperature is concerned, but the water-circulating system has an important part in determining both the yield and the quality of the finished product. There are a number of methods of controlling the water temperature, all of which have some merit. The importance of being able to hold the water at a certain temperature without variation for an indefinite period is not fully recognized by some refiners; but as too high a water temperature means a decreased yield of refined wax, and too low a temperature results in a slower sweating, and in consequence less capacity, prominent refinery engineers consider that the installation of an

automatic temperature controlling device is of decided advantage to the successful operation of a sweating system. The Tagliabue system for the automatic control of the temperature of wax sweaters is shown in Fig. 286.

*Oven Heating Coils.*—The heating coils must be so located that the oven can be kept at a certain temperature without variation, the temperature to be maintained as uniform as is possible. Dry heat only should be used in warming the oven and any steam leakage, no matter how small, should be stopped at once, as open steam in a sweater oven affects both the quality of the wax and the capacity of the plant.

*Piping.*—Both charging and run-down lines should be of ample capacity in order that time lost in charging may be reduced to a minimum and that the "sweat oil" ("foots oil") has a perfectly free outlet.

**Testing Press.**—The determination of the amount of oil and water in scale wax is a test of much significance in practice. For a number of years this test was made with a small lever pump and hydraulic press, which, although very inaccurate, answered the purpose at the time; but when the differences between crude scale, semi-refined, and refined wax were more clearly defined, it became necessary to have a press which would give positive results. The next press to appear on the market was a compound lever press which was a decided improvement over the old hydraulic; but its very complicated construction made it next to impossible to obtain the desired accuracy, and it was eventually superseded by the single lever press.

A testing press of the single lever type is shown in Fig. 287. This press is so designed that a uniform pressure of 1,000 lb. per square inch is applied to the entire area of the test cup and it is said that accurate results may be obtained by the use thereof.

The procedure employed when this testing press is used, is usually along the following lines:

Carefully pulverize and weigh 500 grains of wax. Place two blotters in the bottom of the cup, 5½ in. in diameter: on these blotters place a fine linen cloth and on it carefully spread the pulverized wax, placing another fine linen cloth on top of it, and above the cloth two more blotters. Insert the plunger on top of the blotter and hold under maximum pressure for 5 min. The percentage of oil and water in the sample tested is obtained

by weighing the remaining wax, then multiplying the loss in grains by two (2) and pointing off one decimal place. It may also be found by weighing the increase in the weight of the blotters. For absolute accuracy, the wax should be kept at a temperature of 60°F. throughout.

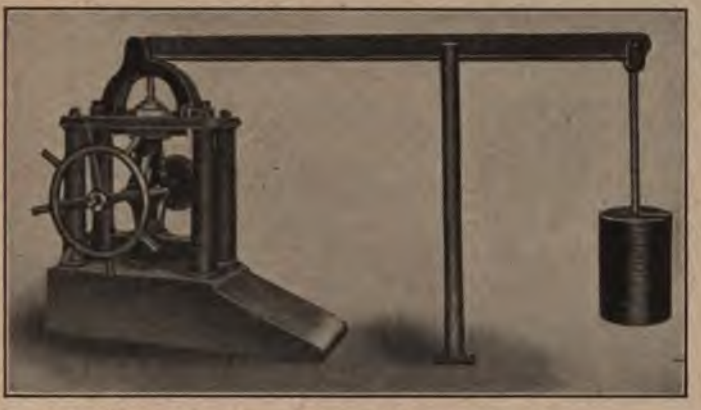


FIG. 287.—Moore's Standard Wax Testing press.

**The Decolorization of Paraffin.**—In the production of paraffin, a requisite treatment is the decolorization of the wax from which the oil has been removed. While there are several methods for obtaining a final product of desired appearance,[1] many refinery engineers consider that when animal charcoal, or "bone-black," is used the best decolorizing results are obtained, both as to complete decolorization and economy of material used for filtration, the "bone-black" being employed continuously over and over again by the use of kilns for revivification. It is used in long vertical cylindrical tanks in rooms heated from 130° to 180°F., and fullers' earth is employed in the same way.

In the apparatus of Robert Sayre Kent, Inc. (Fig. 288), the "wax" in a melted condition and at a temperature of approximately 180°F., is pumped into tanks (11), which have suitable steam coils to maintain the temperature. These tanks are connected by piping to the filters (12), which are filled with "bone-black." The flow is from the tanks into the tops of the filters and downward through the body of bone-black. The elevation of the tanks (11) furnishes sufficient head to enable

[1] In this connection, see the apparatus of RAMDOHR, described in *Dingler's polyt. J.*, **216** (1875), 244.

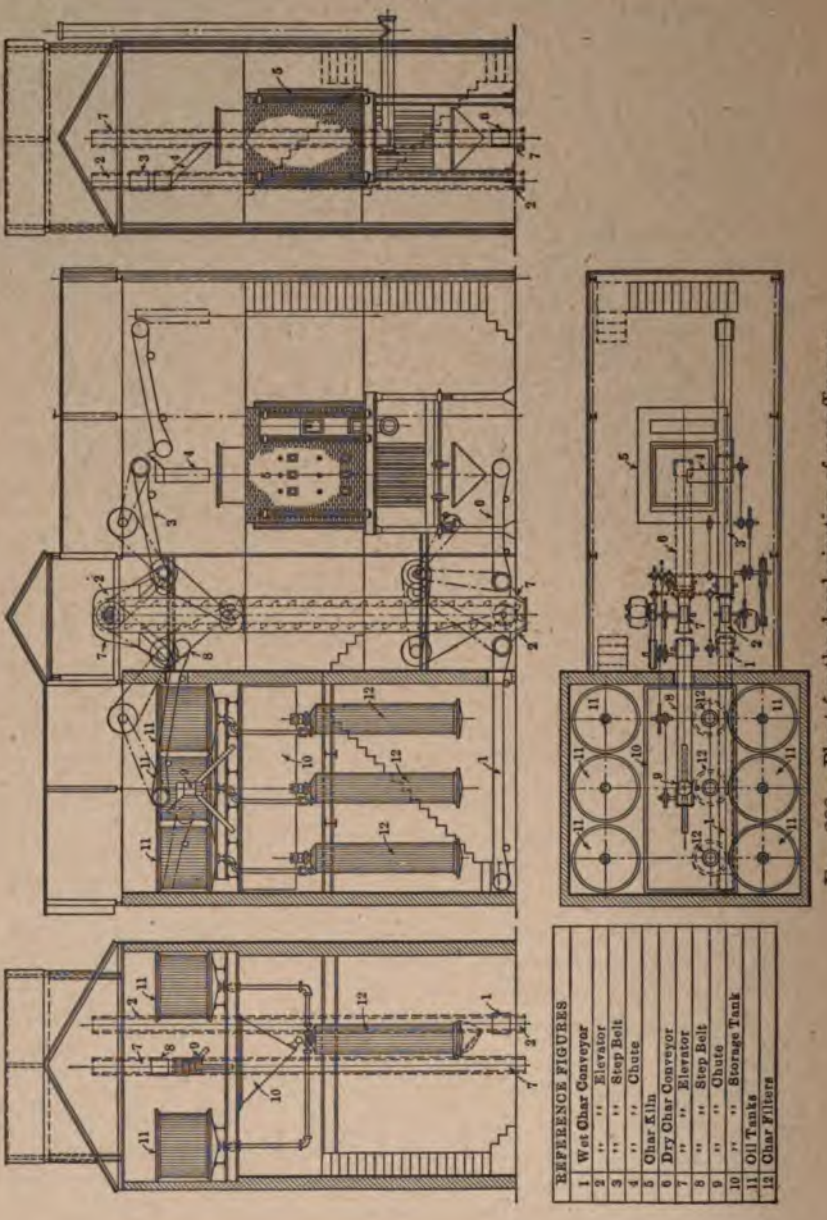



FIG. 288.—Plant for the decolorization of paraffin wax.

the wax to flow through the filters (12). The material is allowed to flow through the filters as long as the product is crystal clear. After this, the "wax" is shut off and the filters are drained. Following this operation, the filter bed is washed throughout with a light perfectly clear oil, which dissolves and carries off all the wax on the "char." This is followed by a steaming process, which removes the light oil and dissolved wax.

This operation is followed with a washing of the "char" in the filter with clear hot water, after which the "char" is ready for removal to the kiln (5). It is very important to keep the "char" flooded at all times with either the wax to be filtered or the oil or water for washing purposes.

The clear melted wax, after coming from the filters, is pumped to another building, where it is run into cool moulds and therein formed into cakes of such sizes as the trade consuming it demands. The wax turns a clear opaque white in chilling, but, if allowed to cool very slowly, would be translucent.

Returning to the kiln (5). These kilns are especially designed with patented features for the so-called "burning" of the bone-black, since, notwithstanding the care in washing, some wax remains in the pores of the "char" and must be removed, as well as the contained organic matter given up by the wax in decolorizing it. These kilns may be arranged to decarbonize the "char," thereby increasing the output of wax and prolonging the life of the "char." This decarbonizing is done, however, by additional apparatus which is not shown. Methods for continuously removing the dust and fine particles resulting from the continual handling back and forth of the "char," are included in the system.

The remaining operations concern the "char" and are the mechanical ones of taking the wet material discharged from the filters (12) onto the belt (1), thence to elevator (2), onto belt (3) to chute (4) into the hopper on top of kiln (5). In kiln (5) the "char" flows from the bottom of the hopper into cast-iron retorts, which are maintained at a suitably high temperature, and through which the "char" flows downward at a fixed rate of speed and in such an intermittent manner that during the progress downward all the "char" is brought into contact with the surface of the retorts.

Provision is made at the top of the retort for the removal of the vapor from any remaining oil or wax in the "char," and also

removal of vapor from the consumed organic matter at the various lower levels of downward movement of the "char" in the retorts.

The finished or so-called "burned char" is discharged into the hopper under the kiln, thence onto belt (6) and into elevator (7), and from there by belt (8) and chute (9) into the storage tank (10), from which tank the filters were originally filled.

**Wax Moulding Press.**—The Moore wax moulding press is similar in construction to a hydraulic filter press in which the rings and filter plates have been substituted by moulds and cooling plates. The sides of the moulds and cooling plates are extended 3 in. above the tops, so that when the "ram" has pushed the movable platen into place, an open gutter or reserve



FIG. 289.—The Moore wax moulding press (Gray's patent).

space is formed along the top of the press over its entire length, which insures communication with every mould. The moulds are made of heavy galvanized iron, in order to prevent corrosion and the appearance of rust in the wax. They are constructed so that each frame will mould two cakes in one operation.

Fig. 289 shows a hundred-plate Moore wax moulding press for machine-moulding paraffin wax by the Gray method. A view of the type of refined wax caking machine in use by several large refiners in the Mid-Continent field is given in Fig. 290.

*Operation of the Moore Moulding Press.*[1]—After the frame is

[1] This press is said to require only one-half of the floor space necessary for the old-style panning system. Moreover, the wax may be moulded in any season, regardless of weather conditions.

set up, the plates and moulds are put in position by first placing a plate next to the stationary platen, and then alternating moulds and plates until the entire equipment is in place, which should bring a cooling plate next to the movable platen. The rubber hose inlet and outlet connections are then made and the press is ready for operation.

To prepare the press for charging, pressure is applied to the hydraulic cylinder, which moves the movable platen along the rods until it comes into contact with the first cooling plate. The pressure is then held at approximately 500 lb. per square inch. A split collar is placed over the rod behind the head, and a tapered



FIG. 290.—The refined paraffin wax caking machine in a Kansas refinery.

key is driven in behind this collar in a keyhole in the rod for this purpose. The pressure may then be relieved, as the key will hold the platen in position. The press is then ready for its initial charge. The water is now turned on, the valve on the wax line is opened and the press is filled as rapidly as possible. It is necessary to have wax enough in the reserve space to make up for the shrinkage due to the chilling. No set rule can be given for the amount, for the operator determines this by practice.

When there is enough molten wax in the press to make perfect cakes, adding a little if necessary, the hot plate is placed on the top of the press. This hot plate is necessary in order to keep the

wax warm while it is shrinking into the moulds, and to prevent the surplus wax from forming on the top of the press after the wax becomes chilled. In 2 to 3½ hr. from the time the press is filled, the wax is ready for removal, the time required depending upon the melting point of the wax and the temperature of the cooling medium. As soon as the wax becomes chilled, the top plate is removed, the surplus wax is scraped off, and pressure is again applied to the cylinder. Finally, the key is driven out, the movable platen is drawn back, and the finished wax is removed by pulling back the cooling plates.

In wax moulded by the Gray method the cake has perfectly square edges and is of uniform thickness throughout, whereas the product of the panning method has all four edges beveled and the top hollow. It is claimed that 260 lb. of wax moulded by the Gray method may be packed in a case 19½ in. wide by 12 in. deep by 37 in. long, while only 216 lb. of pan moulded wax may be put into the same size case.

## MISCELLANEOUS REFINERY EQUIPMENT[1]

**Exchangers.**—When petroleum is distilled continuously, it is usual to transfer the heat from the reduced oil passing from the last still to the oil being charged into the first still of the battery. This transference is effected by passing the hot and cold oil in opposite directions through an exchanger of a system of coils in a steel shell.

Figure 291 gives the specifications for a heat interchanger, 3 ft. in diameter by 18 ft. 6 in. in length.

**Receiving Boxes and Manifolds.**—The pipes which connect the condenser coils with the "tail house," generally located some distance from the condensers, are known as the "running lines." These lines are intercepted in the "tail house" by "look" or "inspection" boxes, in order that the distillate coming from the condenser (the "stream") may be observed by the stillman in charge. The "look boxes" are provided with glass doors and sometimes with cocks for taking samples, and they are connected

[1] The following works may be found to contain suggestive information on mechanical appliances adapted for use in petroleum refineries: PARNICKE'S "Die Maschinellen Hilfsmittel der Chemischen Technik," 2nd ed.; NAGEL'S "The Mechanical Appliances of the Chemical and Metallurgical Industries," 2nd ed.; and NAGEL'S "The Layout, Design and Construction of Chemical and Metallurgical Plants," 1911.

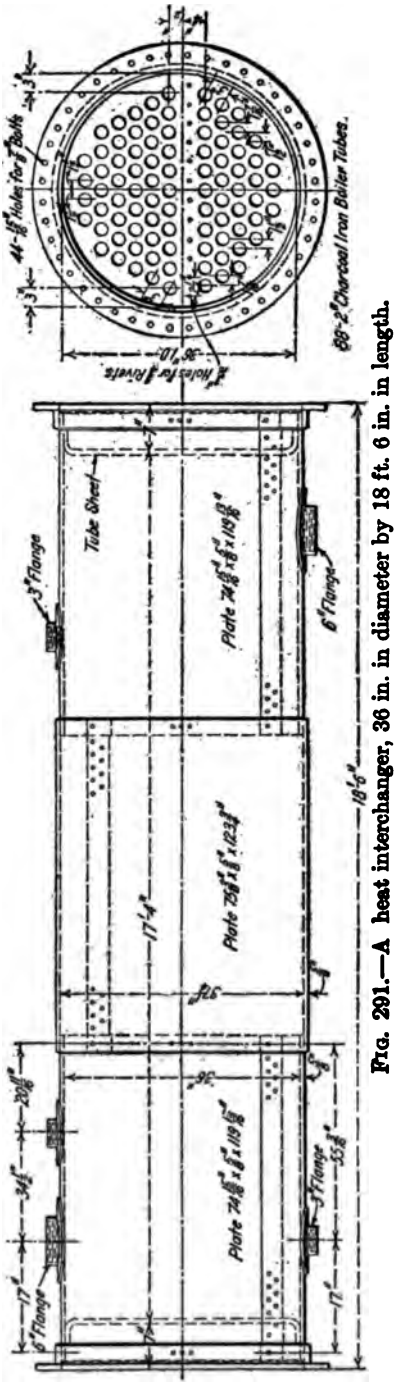



FIG. 291.—A heat interchanger, 36 in. in diameter by 18 ft. 6 in. in length.

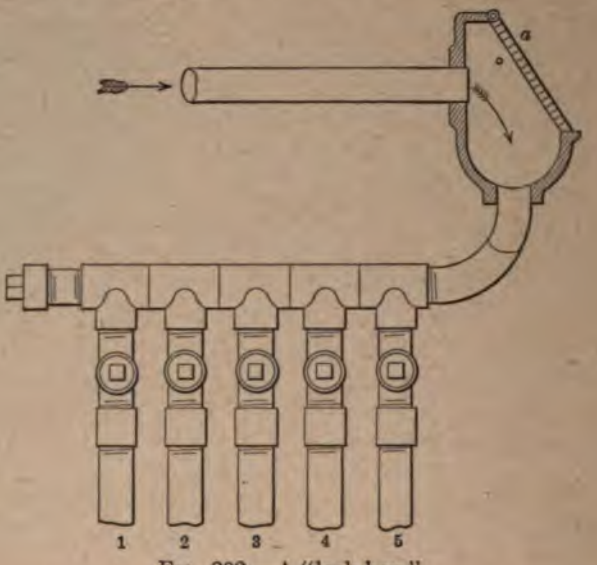



FIG. 292.—A "look box."



FIG. 293.—Receiving house at the reducing stills in the plant of the National Refining Company, Coffeyville, Kansas, showing "look boxes" and manifolds.

at the bottom with manifolds usually having valves, so that the oil may be run into the tank desired.

Figure 292 shows a "look box" equipped with a glass face, *a*, and discharging into the pipes 1 to 5, having cocks arranged so that the distillates may be conducted into any one tank. Such boxes should be gas- and oil-tight, and facilities for sampling the oils and cleaning the glass should be provided. The standard patterns are furnished with flange connections in sizes from 2 in. to 4 in. inclusive.

Figure 293 shows the "look boxes" and manifolds in the receiving house at the reducing stills of the National Refining Company, Coffeyville, Kan.

**Traps.**—In some refineries the vapor pipes from the stills are brought side by side before connection with the condenser, and each

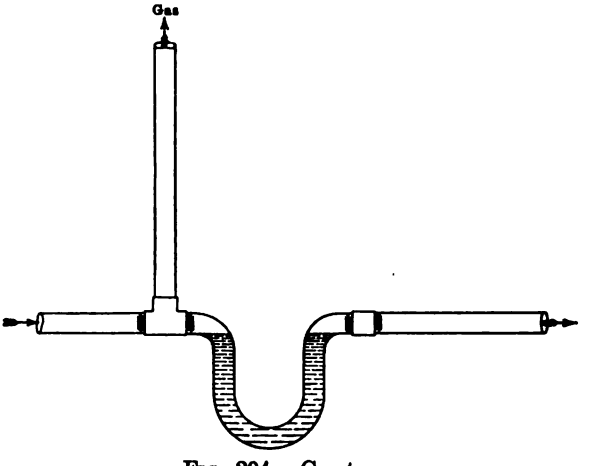



FIG. 294.—Gas trap.

vapor pipe terminates in a gas trap, which allows the liquid portion of the distillate to flow through but checks the gas so that it passes off through a pipe. Fig. 294 exhibits a gas trap of this type.

The "running lines" referred to under receiving boxes, are usually provided with traps for separating the uncondensed gas.

**Spray Nozzles.**—Centrifugal spray nozzles[1] have been employed in petroleum refineries for washing oils at the smallest possible consumption of water. They might be similarly used in the chemical treatment of petroleum products.

[1] For a consideration of liquid atomization, see NAGEL'S "The Transportation of Gases, Liquids and Solids," **1909**, 88–124.

## FIRE EXTINCTION

The extinguishing of fires in oils and in most of the volatile liquids has always been a difficult problem, and where fires of this kind occur the results are frequently very disastrous. The most common extinguishing agent, water, is rather unsatisfactory in the majority of such fires, but it is often the only one available, especially where heroic measures are required. Comparatively recently, however, there have been two or three other materials introduced for use as extinguishers which have shown some promise for dealing with these fires.

Not all fires in volatile liquids are difficult to handle with water. When the liquid is miscible with water, this extinguishing agent can be successfully used. Examples of this kind are denatured alcohol, wood alcohol, etc. Where the liquid is not miscible with water, little or no effect is produced except to wash the burning liquid out of the building where it may be completely consumed, or, if the quantity of oil is small, possibly to extinguish the fire by the cooling effect of a large quantity of water sprayed upon the fire. Sodium bicarbonate and acid solutions are somewhat more effective than water, but even they fail under most conditions. The various grenades containing salt solutions, which were formerly extensively exploited, are said to be practically worthless.

The principles that can be made use of in extinguishing fires in petroleum and its volatile products are: (*a*) to form a blanket either of gas[1] or of solid material (as, for example, foam) over the burning liquid, to exclude the oxygen of the air; or (*b*) to dilute the burning liquid with a non-inflammable extinguishing agent which is miscible with it, such as carbon tetrachloride.[2] The cost of the last-mentioned prohibits its use in the petroleum industry.[3]

[1] In the Coalinga, Cal., fields, where there is frequently but little fire protection, owing to the freedom from lightning, a source of danger in other regions, some tanks are provided with steam pipes below their roofs.

[2] BRESLAUER (United States Patent 990521, Apr. 25, 1911) has claimed a method of extinguishing fires from burning petroleum by adding free bromine to the burning mass. Carbon tetrachloride is used as the solvent for the bromine. In 1875, OMMEGAUCK (*Am. Chemist*, **5**, 292) proposed that *chloroform* be used for extinguishing conflagrations of petroleum.

[3] On the factors affecting fire hazard of oil producing and refining properties. see GUNNISON, *Western Eng.*, Dec., 1915.

In 1882, the use of ammonia for extinguishing petroleum fires was under the consideration of a committee of the Polytechnic Society at Munich.[1] Although the committee was not in a position to recommend the best method of using ammonia or ammonia liquor for this purpose, they expressed the opinion that its employment was well worth the consideration of those who had petroleum stores. Watson Smith[2] pointed out, about the same time, that ordinary ammoniacal gas-liquor of from 5° to 6°Tw. was a cheap and accessible extinguisher for tar refineries, etc.

W. Graff & Co. have patented[3] the use of oxy-halides of sulphur, especially $SO_2Cl_2$, for extinguishing oil fires which cannot be put out by smothering with sand or by the dilution of the burning oil with carbon tetrachloride. The gas is heavier than air and extinguishes the fire by displacing the oxygen.[4]

The use of frothy mixtures for fire extinction was investigated at Wilhelmsburg, near Hamburg, Germany, in 1911, in the presence of officials representing the Prussian Fire Brigade.[5] A solution of caustic soda containing froth-forming ingredients, and a solution containing alum and other chemicals, were mixed together by means of carbon dioxide, which resulted in the production of a foam consisting of bubbles filled with carbon dioxide; the foam was then directed on to the surface of burning petroleum, tar, etc. It was reported that 2 liters of the mixed liquids, when directed on to light petroleum spirit, gave 13 liters of foam, the volume of which diminished by 5 per cent. in 5 min., 14 per cent. in 10 min., 47 per cent. in 30 min., and 76 per cent. in 1 hr. In one series of tests, the plant employed comprised two reservoirs, each of 5-cm. capacity, from which the solutions were delivered to the mixer by two duplex feed pumps at the rate of about 160 liters per minute. The foam was discharged from the mixer through a pipe of 80-mm. diameter, which branched into two pipes of 52-mm. diameter. Fifteen tons of crude benzine were ignited in a brickwork tank of 10 m. diameter; and after the fire had burned for 5 min. in a strong wind, the foam was applied under

[1] *Färb. Must. Ztg.*, **1882**, No. 26.

[2] *J. Soc. Chem. Ind.*, **1**, 351.

[3] German Patent 201393, June 4, **1906**.

[4] *Petroleum*, **4**, 927 (editorial).

[5] *Preuss. Feuerwehr-Beirat, Drucksache*, **1911**, No. 4; *Eng. Min. J.*, **92** (1911), 201; HAMOR, *J. Ind. Eng. Chem.*, **4** (1912), 309; *Eng.*, Jan. 12, **1912**, 52. The Standard Oil Company controls a process which extinguishes fire by means of a chemically formed foam.



FIG. 295.



FIG. 296.



FIG. 297.

FIGS. 295 to 297.—A demonstration of the Erwin fire extinguisher system.



FIG. 298.



FIG. 299.



FIG. 300.

FIGS. 298 to 300.—A demonstration of the Erwin fire extinguisher system.

a pressure of 2 atmospheres. The fire was soon stifled, except for isolated jets of flame near the edge of the tank, and completely extinguished in 6 min.; after 10 min., on removing the layer of foam, the benzine could be immediately relighted. About 1,800 liters of the mixed liquids were consumed. The results seemed to demonstrate that it was advantageous to apply the foam under a low pressure, in order that it might spread quietly over the burning surface. Another test was made on burning tar with satisfactory results.

In 1914, J. B. Erwin and O. R. Erwin were granted patents[1] for a process of extinguishing fires in oil tanks by mixing acid and alkaline carbonate solutions in a chamber separated from but adjacent to the oil tank and allowing the foam to overflow unrestricted from the top of the reaction chamber into the oil tank above the oil.

Figures 295–300 show the operation of the Erwin fire extinguisher system[2] as applied to oil tanks in refineries and in oil fields. In this system foam is automatically discharged, in case of fire, by the fusion of lead links. In tests conducted at Midland, Pa., on a tank 115 ft. in diameter, the fire was completely extinguished in approximately 1 min. and 10 sec. The figures shown herewith are from photographs of a second demonstration, wherein the fire was allowed to continue for 5 min. with the automatic features removed from the system; at the expiration of that time, the fire extinguisher was discharged and extinction resulted in approximately 45 sec. after it began to operate.

According to the process of E. Bartels,[3] fires in oil tanks or boats are extinguished by mixing acid, sodium bicarbonate and extract of licorice root in a chamber near the tank in which the fire is to be extinguished, and conducting the mixture produced to the point of use by the pressure of the gas which is generated from the mixture.

The extinction of fires in oils has been discussed by Barrier in several contributions.[4] He maintains that sawdust, moist or

[1] United States Patent 1085805, Feb. 3, **1914**; English Patent 2142. Jan. 27, **1914**.

[2] Published by courtesy of the Treadwell Construction Co., Midland, Pa.

[3] United States Patent 1115887, Nov. 3, **1914**.

[4] *Met. Chem. Ind.*, **12**, 99; *Western Eng.*, **3** (1913), 476; and *Metal Ind.*, **11**, 119. See also Bowie, *Western Eng.*, **4** (1914), 105.

dry, soft or hard wood,[1] is superior to sand for extinguishing fires in tanks of inflammable liquids; that carbon tetrachloride is useful as an extinguisher, but that its anesthetic properties prevent its general use; and that frothy mixtures will prove to be the most efficient.

Leakage aboard naval vessels using oil fuel has resulted in the demand for an extinguishing material which will flow into corners. Williams[2] states that the most promising system is the use of a foam mixture composed of glue,[3] glucose, sodium bicarbonate, salicylic acid, aluminum sulphate, sulphuric acid, and water. The aluminum sulphate and sodium bicarbonate produce carbon dioxide; the glue renders the liquid viscous and produces foam; the salicylic acid is a preservative for the glue and glucose, which in turn acts as a stabilizer to retard the precipitation of aluminum hydroxide.

[1] Ten pounds of sodium bicarbonate per bushel of sawdust is an improvement.

FAGAN (United States Patent 1148763, Aug. 3, **1915**) extinguishes oil fires by blowing onto the surface of the burning oil a light powder such as sawdust or cork, or other material which will float on the oil, the particles of which are coated or mixed with a fire-extinguishing liquid, such as the reaction products of sodium bicarbonate and sulphuric acid. For other fire-extinguishing compositions, see W. W. WALKER, United States Patent 1161090, Nov. 23, **1915**; and STANZIG and KÖNIG, English Patent 16967, July 17, **1914**.

[2] *Eng. News*, **67**, 921.

[3] Extract of licorice root is preferable on account of its unobjectionable odor. Quillaia bark and saponin are other foam-forming substances which can be employed.

# CHAPTER XV

## HYGIENIC CONSIDERATIONS

Crude petroleum, as well as many of its distillation products, may be a source of considerable cutaneous and other irritation, especially among workmen who are unhygienic in their habits. Thus Bertenson,[1] who, in 1895, made a special study of the workmen employed at the petroleum wells of Tartary and Persia, found that, among 8,465 workmen, 1,216 had various skin eruptions and 1,475 suffered from respiratory diseases. In the United States, however, the modern methods of treating and transporting petroleum and its products, now largely carried out by mechanical means, have reduced the hazards to a minimum. Cleaning out petroleum stills, agitators and tanks is one of the main risks. In general, the lightest products of distillation are the most toxic; they possess an anesthetic action and may decompose or liberate hemoglobin from the red blood cells. Experimentally, in animals, the inhalation of these products gives rise to congestion of the lungs and kidneys.[2]

In 1914, Hayhurst[3] investigated the refining of petroleum in four Ohio establishments. He found that practically everything was handled by the most modern devices, whereby most

[1] *D. Vierteljahrsschr. f. öffentl. ges.-Pfl.*, **30** (1898), 315; *Gorn. Jurn.*, **3** (1897), 216.

[2] POINCARÉ (*J. Pharm.*, (5), **7**, 290-291) kept animals continuously in an atmosphere which was frequently charged with the vapor of petroleum. The effects observed were principally greater frequency and amplitude of the respiratory movements, a diminution of the cardiac movements, and a greater intensity of the beating of the heart. This was accompanied by cutaneous irritation, and a general tendency to sleep and languor. Guinea-pigs finally succumbed under the treatment. The autopsy showed general congestion. According to POINCARÉ, workmen engaged in the distillation of petroleum complain of heaviness in the head and great irritation of the mucous membrane of the nose. On the toxicology of petroleum, see LEGIUDIC and TURLAIS' "Recherches sur la Toxicité du Pétrole et quelques-unes de ses Actions Physiologiques," Paris, **1915**. On the effects of breathing air charged with petroleum vapor, see *Chem. News*, **47**, 191.

[3] "Industrial Health-hazards and Occupational Diseases in Ohio," Ohio State Board of Health, **1915**, 339.

materials were confined and moved from place to place by means of pumps and piping, so that the workers had little to do outside of watching the machinery and of opening, cleaning and closing of stills and tanks. Hoods and exhausts were generally present where required. General health-hazards were limited, although many of the workmen were exposed to inclemencies of the weather and long hours prevailed. It was reported that cases of sore eyes,[1] due to the action of the products, were not infrequent; that the workers complained of the effects of hydrogen sulphide,[2] sulphur dioxide and sprays of sulphuric acid in some refineries; and that dust was bad in a desulphurizing process using a mixture of copper, lead and iron oxides. Hayhurst states that "the development of 'paraffin cancer' has been found to occur in the handlers of the crude heavy products, as well as considerable dermatitis and acne."

### PETROLEUM REFINERY HYGIENE

**Effect of Petroleum on Health.**—Industrial poisoning in the petroleum industry is attributable to the vapors evolved from crude petroleum or its products. Such poisoning may occur in the production of crude petroleum, in storage and transport (in badly ventilated tanks on board ship, and in entering petroleum tanks), in the refinery in cleaning out petroleum stills, agitators, and tanks, and in emptying out the residues. Further cases occur occasionally from the employment of benzine in chemical cleaning. In addition to poisoning, the injurious effect of petroleum and certain of its products on the skin is to be considered. Opinion is unanimous that the injurious action of mineral oil in this respect is limited to petroleum products with high boiling points and to residues.

Official Prussian statistics show that the general health of petroleum workers is favorable. These statistics related to 1,380 persons, of whom 43 were suffering from symptoms attributable to their occupation. Of these 43, nine only were cases of poisoning, the remainder being all cases of "petroleum acne."

[1] Three positive cases of conjunctivitis are mentioned (*ibid.*, 368).

[2] The still gases from refineries handling Gulf field crude petroleums (see p. 71) contain sulphur compounds and have also been the cause of complaint. There are instances on record of the death of animals from the inhalation of these gases and of the prevalence of "pink eye" among workmen exposed to their action. Care is now exercised in practice and the still gases are confined as much as possible.

The conditions in French refineries appear to be satisfactory from statistics collected in the years 1890–1903. Eighteen cases of "petroleum acne" were reported, eleven of which occurred at the paraffin presses, five in cleaning out the still residues, and two in filling vessels.

The conditions have been less favorable in the Russian petroleum industry.[1] The workers at the naphtha wells suffer from acute and chronic affections of the respiratory organs. This is particularly the case with those who cover the wells with cast-iron plates to enable the flow of naphtha to be regulated and conducted into the reservoirs. In doing this they inhale naphtha spray.

Lewin[2] has referred to cases of severe poisoning with fatal issue among American workers employed in petroleum tanks. One man who wished to examine an outlet pipe showed symptoms after only 2 min. Weinberger has described severe poisoning of two workers engaged in cleaning out a vessel containing petroleum residuum.[3]

Respiratory affections were rarely found among workmen engaged at the Carpathian wells, but poisoning symptoms involving unconsciousness and cerebral symptoms occurred frequently.

These experiences undoubtedly indicate differing physiological effects of different kinds of petroleum. This is supported by the view expressed by Sharp[4] that different kinds of American petroleum have different effects on the health of the workers, which can be credited on account of the variations in composition. Thus where natural heavy oils are obtained, asphyxia from gas is unknown, although transient attacks of headache and giddiness may occur; whereas in cases where light oils are obtained, suffocative attacks may not be infrequent. It is stated that some petroleum products irritate the respiratory passages, while others affect the central nervous system.[5]

Scheller and Stauss[6] reported that the presence of carbon monoxide in the gases resulting from the distillation of petroleum residues containing paraffin was the cause of the death of a work-

[1] BERTENSON, *loc. cit.*

[2] *Arch. path. anat.*, **112** (1887), 35.

[3] *Wien. Med. Halle*, **4** (1863), 40.

[4] *Med. News*, Aug., **1888**, 150.

[5] FELIX, *D. Vierteljahrsschr. f. öffentl. ges.-Pfl.*, **1872**, 226.

[6] *Petroleum*, **8**, 849. Carbon monoxide has also been the cause of death in drilling operations (SHREWSBURY, *Analyst*, **37**, 486).

man engaged in repairing the interior of a still which had been employed for such distillation. Experiments with mice showed that the vapors of benzine had an injurious effect, but that the mice were readily restored to life by means of air or oxygen. Placed in the gases from the distillation of lubricating oil, mice died quickly. An analysis of these gases gave the following results:

| | Soon after beginning of distillation, per cent. | After 1 hr., per cent. | After ⅓ had been distilled, per cent. | End of distillation, per cent. |
|---|---|---|---|---|
| Carbon dioxide..... | 3.5 | 6.0 | 3.0 | 2.5 |
| Carbon monoxide..... | 16.0 | 8.5 | 16.5 | 10.0 |
| Unsaturated hydrocarbons..... | 3.5 | 12.0 | 3.5 | 16.5 |
| Saturated hydrocarbons..... | 77.0 | 73.5 | 77.0 | 71.0 |

The amount of carbon monoxide in the gases from the distillation of crude oils from different localities varied from 1.1 to 8.0 per cent. The formation of carbon monoxide was assumed by Scheller and Stauss to be caused by the decomposition of the acids and esters present in the residues. No trace of carbon monoxide was found in the gases when the residues were treated, before distillation, with alkali and washed well. Refined machine oil yielded no carbon monoxide. The introduction of air during distillation caused the formation of carbon dioxide in the gases from the distillation of a refined machine oil, which, in the absence of air, gave no carbon monoxide.

There is authoritative mention in the literature of the occurrence of cases of poisoning in the refining of petroleum, from inhalation of the vapors of benzine and gasoline. Fatal cases have been recorded in badly ventilated workrooms in which the products of distillation are collected. Workmen constantly employed in these rooms may develop chronic poisoning, which is reported also in the case of women working with benzine. Intoxication has sometimes been observed among the workmen employed in cleaning out tank cars in which mineral oils are shipped.

Foulerton[1] has described severe poisoning in a workman who had

[1] *Lancet*, **1886**, 149.

climbed into a petroleum reservoir, and two similar cases from entering naphtha tanks in England are given in the Report of the Chief Inspector of Factories for 1908. Two fatal cases were reported by the Union of Chemical Industry in Germany in 1905, in connection with naphtha stills. Such accidents are hardly possible, except when, through insufficient disconnection of the still from the further system of pipes, noxious distillation products pass backward into the opened still where persons are working. Ordinary cocks and valves, therefore, do not afford sufficient security. Thus, according to Rambousek,[1] several workers engaged in repairing a still were rendered unconscious by gases drawn in from a neighboring still, and recovered only after oxygen inhalation. The gases evolved during the distillation of "sulphur crudes," such as the petroleums of the Lima-Indiana and Gulf fields, should always be confined as much as possible.[2]

Gowers described a case of chronic poisoning following on frequent inhalation of gases given off from a petroleum motor, the symptoms being slurring speech, difficulty of swallowing, and weakness of the orbicularis and facial muscles. He believed this to be petroleum gas poisoning (from incomplete combustion), especially since the symptoms disappeared on giving up the work, only to return on resuming it again.[3]

Recent literature[4] mentions the occurrence of "petroleum eczema" in a firebrick and cement factory. The workers affected had to remove the bricks from moulds on to which petroleum had dropped. An eczematous condition was produced on the inner surface of the hands, necessitating abstention from work. The pustular eczema in those employed only a short time in pressing paraffin in petroleum refineries is referred to as a frequent occurrence. Practically all the workers in three refineries in the district of Czernowitz were affected. The view that it is due to insufficient care in washing is supported by the report of the factory inspector in Rouen, that with greater attention in this matter on the part of the workmen marked diminution in its occurrence followed.

The injury to health produced by crude petroleum and its

[1] "Industrial Poisoning," **1913**. On dangers of distilling, see also *Chem. News*, **6**, 312.

[2] See p. 609.

[3] *Ramazzini*, **2** (1908), 226.

[4] RAMBOUSEK, *op. cit.*

products is, then, of two kinds.[1] Direct contact with liquid petroleum and the semi-liquid and solid residues after distillation may cause local injury to the skin. Inhalation of the volatile constituents of crude petroleum may produce symptoms affecting mainly the central nervous system; they have, moreover, a markedly irritating effect upon the mucous membrane of the respiratory organs. These substances show clearly that the hydrocarbons boiling at low temperatures act as nerve poisons, whereas those boiling at higher temperatures produce a local irritant effect.

**Symptoms.**—The symptoms of petroleum poisoning[2] may be either acute or chronic.

*Acute Type.*—The acute form, known sometimes as "petroleum intoxication," may be very acute and promptly fatal. The victim is seized with sudden weakness of the legs and dyspnea. He may fall down in coma and die of asphyxia on entering a non-ventilated, partially emptied reservoir. Korjenevski reported the case of a man who worked for a day at a petroleum well, when he was seized with hemoptysis, hematemesis, melena and delirium, and died the following day.[3] The comatose cases are not always fatal, and a patient may remain in coma for a week and yet recover. In very mild cases some of the workmen complain of vertigo, headache, nausea, bronchitis and mental depression, but others have a feeling of elation and rather like the intoxication.

*Chronic Type.*—Chronic poisoning causes vertigo, fullness and throbbing of the head, cough and dyspnea, anemia, general nervousness, hallucinations, and loss of memory. There may be also attacks of syncope and loss of consciousness. The effects

[1] In this connection, see BERTENSON, "Die Naphthaind. in sanit. Beziehung," *Vortrag auf dem XII. Intern. Aerztekongr. in Moskau*, **1897**; and *D. Vierteljahrsschr. f. öffentl. Ges.-Pfl.*, **30**, 315; BURENIN, "Die Naphtha u. i. Verarb. in sanit. Beziehung," Petrograd, **1888**; LEWIN, *loc. cit.*; SHARP, *loc. cit.*; SAMUEL, "Verg. in Petroleumtanks," *Berl. klin. Wochenschr.*, **41**, 1047; FOULERTON, *Lancet*, **1886**, 149; MABILLE, *Revue d'Hyg.*, **18**, No. 3; *Concordia*, **1909**, 105.

On the effects of petroleum vapor in air, see POINCARÉ, *Compt. rend.*, **96** (1883), 353; and WEINBERGER, *Wein. Med. Halle*, **4** (1863), 40.

[2] It should be understood that occupational diseases in the petroleum industry are not necessarily extrinsic poisonings, but are just as much the result of intrinsic poisonings and toxins. However, extrinsic poisoning by petroleum is regarded above as specific.

[3] See *Vratch*, **1887**, 350.

vary with different persons, some being excited and others calmed, as if they were under the influence of opiates.

The skin affections usually take the form of inflammation of the hair follicles (acne), eruptions with characteristic formation of vesicles, and pimples and pustules which precede the deep-seated formation of ulcers, abscesses, etc.[1]

**Prophylactic Measures.**—As crude petroleum and the higher fractions distilled from it may affect the skin injuriously, careful cleanliness on the part of the workmen should be enjoined. Workmen exposed to the influence of gases escaping from oil wells should be equipped with breathing apparatus (smoke helmets); this applies also to those who are obliged to enter stills and other apparatus connected with the treatment of petroleum in the refinery.

In the treatment of petroleum with sulphuric acid, sulphur dioxide is evolved, and it may constitute a distinct danger to the workers. This process should preferably be carried on in closed vessels furnished with mechanical stirrers or compressed-air agitators, and the gases evolved should be taken care of.

Petroleum tanks should be thoroughly aired before they are cleaned and should be entered only by workers equipped with breathing apparatus. Special care should be exercised at all pumps, reservoirs, tanks, conduits, etc., to prevent leakage and accumulation of vapors where they might be inhaled. The dangers of electrical short-circuits, friction sparks and flames should be understood by the workers and guarded against.[2]

Workmen should take frequent hot-water baths, using soap freely, and should have their overalls washed often. Good first-aid arrangements should always be provided. As a rule, in American refineries the workmen are quite well protected, so that serious poisoning is uncommon.[3]

[1] See ESPAGNE and SARDUN, *Gaz. hebd. Sci. Méd. Montpellier*, **13** (1891), 193. The occurrence of skin affections in the petroleum industry has been reported by certain observers, especially among those employed on the unpurified oils. Eruptions on the skin from pressing out the paraffin and papillomata (warty growths) in workers cleaning out the stills are referred to by several writers (ARLIDGE, *Rev. d'Hyg.*, **1895**, 166; MITCHELL, *Med. News*, **1888**, 152; *Ann. Hyg. publ.*, **24**, 500; and NEISSER, *Uebers. Gew.-Hyg.*, **1907**, 96).

[2] It should be noted here that explosions in agitators are usually attributable either to such causes as electrical short-circuits or sparks caused by the friction of hard surfaces, or to the flow of hydrocarbon vapors in the direction of a neighboring flame.

[3] See HAYHURST, *loc. cit.*

## LIGHT PETROLEUM DISTILLATES

Acute poisoning from inhalation of the vapor of benzine begins with headache, sickness, and attacks of giddiness resembling alcoholic intoxication. If very much has been inhaled, the patient quickly becomes unconscious, with occasionally muscular tremors, convulsions, difficulty in breathing, and cyanosis. In cases of poisoning by inhalation of crude petroleum vapor, these symptoms may be complicated by coughing, intense inflammation of the mucous membrane of the respiratory organs—congestion, bronchitis, blood-stained expectoration, and inflammation of the lungs. In workmen who frequently remain long in an atmosphere filled with benzine vapor, further symptoms of chronic benzine poisoning show themselves—mental hebetude, pains in the limbs, trembling, weakness of the muscles, and other disturbances of the nervous system; in such cases these may really be signs of continued attacks of acute or subacute poisoning; many benzine workers are anemic.[1] 0.05 gram of benzine per liter of air is poisonous, while 0.02 gram per liter causes local symptoms.

According to Jaffe,[2] benzine is chiefly, if not entirely, excreted through the lungs. Where the resorption and therefore the excretion through the lungs proceeds rapidly, extensive pulmonic hemorrhages are found. The resorption from the stomach proceeds slowly, leading to extensive necrotic and inflammatory changes of the lungs.

Thompson[3] states that the symptoms of acute "naphtha poisoning" resemble those of benzol poisoning. Headache, vertigo, dyspnea, palpitation, nausea and vomiting are chiefly complained of. There may be mental confusion and death may result from hemorrhage from the respiratory mucosa or paralysis of the heart. Sometimes there are acute hysterical attacks with insomnia. Autopsies in cases in which the patient

[1] On benzine poisoning, see ALPAUGH, *Ohio Pub. Health J.*, **6** (1915), 512; BOX, *Brit. Med. J.*, **1908,** 807; *Z. Gew.-Hyg.*, **1906,** 515; **1907,** 157; **1908,** 333; BÜRGI, *Korr. Schweiz. Aerzte,* **36** (1906), 350; DORENDORF, *Z. klin. Med.*, **1901,** 42; FINLAYSON, *Brit. Med. J.*, **1903,** 546; and SCHÄFER, *Hamb. Gew.-Insp.-Arb., u. Sonderber.*, **1909,** 7. On the physiological action of petroleum ether, see GEORGES, *Compt. rend.*, **58** (1864), 1192; and FELIX, *loc. cit.*

[2] *Munch. med. Wochschr.*, **51,** 175.

[3] "The Occupational Diseases," **1914,** 334. Cf. KOBER and HANSON'S "Diseases of Occupation," **1916,** 136.

dies after a few days, so that morbid changes have had time to take place, reveal fatty degenerations of the most important viscera, heart, kidneys and liver. The same authority states that chronic "naphtha poisoning" results in chronic bronchitis, tinnitus aurium, vertigo, headache, and mental dullness.

**Treatment.**—The treatment of acute benzine poisoning consists in oxygen inhalation, with simultaneous artificial respiration. Treatment of chronic derangement of health is symptomatic.

**Uses Involving Risks.**—Girls employed in glove cleaning and rubber factories are described as having been poisoned by benzine.[1]

Poisoning of chauffeurs is referred to by several writers.[2] In fact, recent literature[3] tends to show a marked increase in the number of cases of poisoning from greater demand for benzine as a motive power for vehicles. Such cases have been observed in automobile factories, and have been attributed by some to the hydrocarbons of low boiling point present as impurities in benzine.

Rambousek[4] cites the following cases of poisoning:

1. A worker in a paraffin factory had entered an open benzine still to scrape the walls free of crusts containing benzine. He was found unconscious and died some hours later. It appeared that he had been in the still several hours, having probably been overcome to such an extent by the fumes as to be unable to effect his escape.

2. A night worker in a bone extracting works having turned on the steam, instead of watching the process, fell asleep on a bench. In consequence, the apparatus became so hot that the solder of a stop-valve melted, allowing fumes to escape. The man was found dead in the morning.

3. In a carpet cleaning establishment three workers lost conscious-

[1] DORENDORF, *Z. klin. Med.*, **1901**, 42.

[2] *Brit. Med. J.*, **1903**, 546; **1908**, 807; THOMPSON, *op. cit.*, 333. A number of cases of chauffeurs who were overcome by gasoline fumes while cranking motor cars in small unventilated garages, are recorded in the literature. They experienced vertigo, fainting and nausea, and one passed into coma, from which it was difficult to arouse him. The symptoms may not develop until the men go into open air. On "petromortis" and contamination of air by automobile exhausts, see APPELBACH, *Heat. Ventil. Mag.*, **13** (1916), 48.

[3] *Z. Gew.-Hyg.*, **1907**, 157.

[4] RAMBOUSEK'S "Industrial Poisoning," **1913**, 63.

ness and were found senseless on the floor. They recovered on the inhalation of oxygen.

One further case reported from the instances of benzine poisoning collected recently[1] is worthy of mention.

A worker in a chemical factory was assigned to clean a still capable of holding 2,500 liters of benzine, which contained the remnants of a

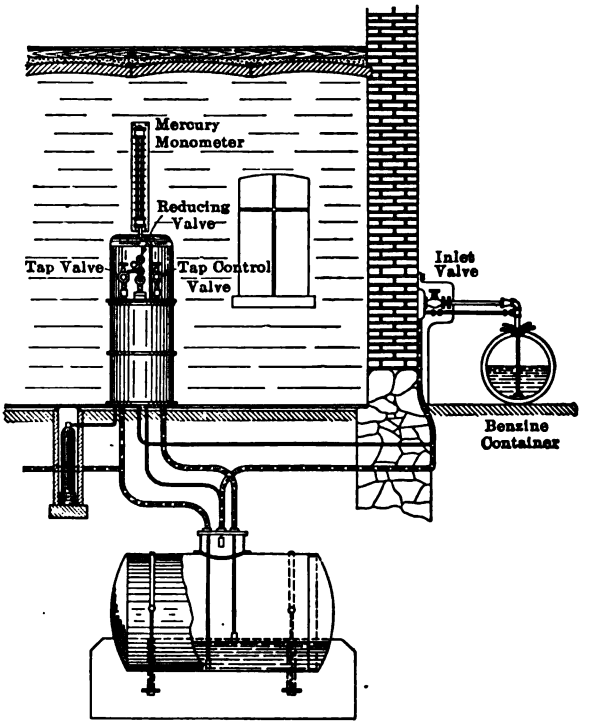



FIG. 301.—Cross-section of an automobile garage provided with safety devices for minimizing risk from fire.

The taps can be placed in any part of the building as desired. The amount of gasoline contained in the storage-tank is ascertained by reference to an automatic gauge in the room where the liquid is used. The same scheme may be applied to industries wherein benzine is used.

previous filling. As soon as he had entered the narrow opening, he became affected and fell into the benzine; he was carried unconscious to the hospital, his symptoms being vomiting, spastic contraction of the extremities, cyanosis, weak pulse, and loss of reflexes, which disappeared an hour and a half later.

[1] WICHERN, *Z. Gew.-Hyg.*, **1909**, Nos. 3 and 4.

The men who clean out large gasoline tanks may suffer from vertigo and asystole, and the workmen employed in cleaning distilling apparatus may be acutely poisoned by inhaling naphtha vapor. Most workmen, however, become accustomed to the irritation, which at first may cause bronchial catarrh.

Naphtha vapor is liberated in the preparation of waterproof cloth, the rubber industry, dyeing, and a variety of cleaning processes, although the employment of naphtha for the latter purpose has been supplemented to a considerable extent by other substances. In the manufacture of rubber cloth, rubber shoes and patent leather, in which the rubber is spread over a prepared surface, naphtha is used extensively as a solvent. It is employed also as a bath to disinfect clothing worn by those having infectious diseases, and as a solvent for metal polish. A case of dermatitis and eczema of the hands caused by polishing metal bedsteads was reported to the New York State Bureau of Labor in 1912; the polish was dissolved in naphtha.

In Germany, the following accidents occurred from the use of benzine during 1914:

| Character of accidents | Persons hurt | | | |
|---|---|---|---|---|
| | Total No. | Slightly | Seriously | Deaths |
| Chemical cleaners and dyers | 6 | ..... | 4 | ..... |
| Druggists, etc | 11 | ..... | 3 | 1 |
| Benzine in technical use and transport | 62 | 27 | 56 | 76 |
| Benzine in drains and sewers | 3 | ..... | 2 | ..... |
| Benzine in automobile traffic | 104 | 38 | 55 | 27 |
| Benzine for illumination | 10 | 1 | 4 | 1 |
| Benzine for household uses | 42 | 7 | 30 | 10 |
| Narcosis from benzine vapors | 8 | 3 | ..... | 5 |
| Totals | 246 | 76 | 154 | 120 |

There was an increase of accidents from the use of pocket lighters, and the number of explosions of benzine used for automobiles nearly doubled. In the report of these accidents,[1] emphasis was laid on the fact that non-inflammable substitutes

[1] *Z. angew. Chem.*, **28** (1915), 73.

for benzine exist which are suited for most uses except power production and illumination.[1]

**Prophylactic Measures.**—Apparatus containing petroleum and benzine should, as far as possible, be closed in and air-

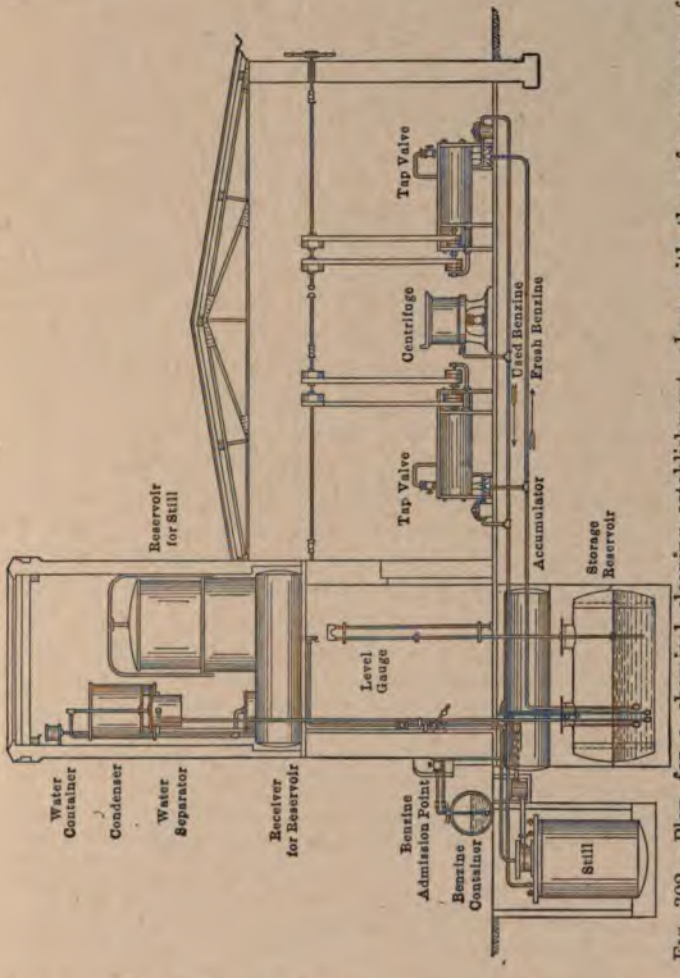



FIG. 302.—Plan for a chemical cleaning establishment, along with the safer recovery of benzine through distillation.

The benzine, or other inflammable liquid, flows out of the taps under the normal pressure of protecting gases. On the storage of benzine, see POLACK, *J. Gasbel.*, **49** (1906), 337; and EFFENBERGER, *idem*, 689. On the Martini-Hüneke apparatus for washing with benzine, see *Chem. App.*, **2** (1915), 281.

tight (as, for example, in the extraction of fat from bones, in the rubber industry, and in chemical cleaning establishments); where benzine vapors develop they should be immediately drawn

[1] On this point, see also *Z. angew. Chem.*, **20** (1907), 241, wherein carbon tetrachloride *vs.* benzine is discussed.

away by suitably applied exhaust ventilation. On account of the danger of fire,[1] this is necessary in chemical cleaning establishments.[2]

German regulations for benzine extraction plants are contained in the Prussian Ministerial Decree, dated Jan. 5, 1909, for benzine extraction works, and also in that of Aug. 3, 1903, for dry-cleaning premises, to which last were added "Directions for Safety," containing important regulations as to risk from fire. The following points are of interest: Care is to be taken to provide and maintain exhaust ventilation directly across the floor. The air, however, must not be allowed to pass near any fire. Drying rooms especially are to be lofty and airy, and separated from other workrooms. In factories with mechanical power the authorities may require provision of artificial ventilation for the drying rooms. Washing machines, centrifugal machines, and benzine rinsing vessels should be furnished with well-fitting covers to be removed only for such time as is absolutely necessary for putting in and taking out the articles to be cleaned, shaken, or rinsed. The vessels named are to be examined as to their imperviousness, at least once every quarter, by a properly qualified person. The condition in which they are found is to be noted in a book to be shown to the factory inspector and police authorities on demand. Lastly, wherever possible, the substitution for benzine of other less poisonous and less inflammable substances should be considered.[3]

[1] On the causes of the ignition and explosibility of benzine, see LUCCHINI, *Ind. chim.*, **12**, 65 and 81. Neither the gases from nor the vapors of petroleum have yet been shown to be spontaneously inflammable (*Chem.-Ztg. Repert.*, **24**, 295). On various apparatus for the fire-proof storage of benzine, see STRACHE, *Z. Ver. Gas Wasserfachm.*, **55** (1915), 191 and 209.

[2] To prevent the explosions which result from the generation of electricity in wool and silk when these materials are moved about while saturated with light petroleum, FISCHER (*Leipziger Färber- u. Zeugdr.-Ztg.*, **49** (1900), 287) makes an addition to the latter of 1 per cent. of magnesium oleate.

On extinguishing petroleum fires, see p. 776.

[3] BERTENSON, "Die Naphthaindustrie in sanit. Beziehung," *Vierteljahrsschr. f. öffentl. Ges.-Pfl.*, **30**, 315; KORJENEVSKI, *Vratch*, **1887**, No. 17; BURENIN, "Die Naphtha und ihre Verarbeitung in sanit. Beziehung," **1888**; MABILLE, *Rev d'Hyg.*, **18**, No. 3; *Ber. der Berufsgen. f. chem. Ind.*, **1905**; *Ber. der preuss. Gew.-Insp.*, **1904**; KLOCKE, *Z. Gew.-Hyg.*, **1908**, 379; "Benzinersatz," *Z. Gew.-Hyg.*, **1906**, 248; **1908**, 384.

### PARAFFIN

Melted paraffin may evolve vapors which, when inhaled, give rise to headache, vertigo, anorexia, nausea, vomiting, gastralgia, enteralgia, diarrhea, thoracic oppression, cold sweats and lassitude.

Locally paraffin irritates the skin, especially the sebaceous glands, and a few cases of epithelioma have been ascribed to its use. It may give rise to acne-like skin inflammations ("paraffin eczema") and pustular eczema of the hands, face and ears.[1]

Workmen handling or using paraffin[2] should protect the hands, which should be cleansed frequently in alkaline solutions. Rubber gloves should be worn when possible.

### ASPHALT

Workers in asphalt who stand over the melting pots are exposed to the irritating vapors which arise. They may acquire catarrhal conjunctivitis and bronchitis, and a uniform eruption may appear over the entire body, which assumes a yellowish color. Four cases of asphalt poisoning have been reported by Kemp.[3]

Truc and Fleig[4] have recently investigated the lesions produced by the dust and vapor of asphalt. They found that asphalt dust caused a severe affection of the cornea in workmen exposed thereto: Pure asphalt dust produced blepharo-conjunctivitis, corneitis (interstitial or ulcerating), episcleritis and iritis in dogs and rabbits; and the experimental injuries were the same as in man. Differences in the intensity and rapidity of appearance of the effects were observed; these were attributed, among other things, to individual characteristics and to the nature of the dust. Asphalt vapor caused mild inflammation of the conjunctiva in rabbits. In general, it was found that injurious effects depended more upon chemical than upon bacterial and mechanical factors.

[1] On skin diseases among workers in paraffin, see BREMOND, *Rev. d'Hyg.*, **1895**, 166; CHEVALLIER, *Ann. Hyg. Publ.*, **1864**; DERVILLE and GUERMONPREZ, *Ann. derm.*, **1890**, 369; LEWIN, *loc. cit.*; MITCHELL, *Med. News*, **53**, 152; and RAMBOUSEK, *Concordia*, **1910**, No. 6.

[2] For example, in waterproofing (protecting the containers in which explosives are packed; spraying dynamite cartridges, etc.) and in the insulation of electric systems, but particularly in paraffin pressing in the refinery.

[3] *Z. med. Beamte*, **1903**, 271.

[4] *Arch. opth.*, **133**, 538 and 593; *Zentr. Biochem. Biophys.*, **16**, 222.

In this connection, mention may be made of the effects of tar roads upon animals and vegetation.

It has been stated that the dust from tarred roads has appeared to be more irritating to the eyes than that from untarred roads, and in 1910 an investigation of this matter was conducted in France by Truc and Fleig.[1] It was ascertained that dust from untarred roads had only the slightest effect when sprinkled on the eyes of animals, and that dust from old tarred roads, from which the coating had more or less disappeared, gave little different effects. However, dust from old tarred roads with a well-preserved surface occasioned conjunctivitis and other lesions, while dust artificially produced from such roads gave still more severe effects. Notwithstanding the fact that bituminous vapors are said to have only a slight action upon the eye,[2] the investigations of Truc and Fleig seemed to show that the results corresponded to the proportion of tar, as well as to the mechanical irritation produced by the dust and to the germs present. Concerning these observations, Baskerville has remarked[3] that while bituminous dust may rapidly produce various lesions in the eyes, and may leave leucoma, the condition of the eye and the action of sunlight are both predisposing causes. He noted that the experiments which have been conducted on these points do not, moreover, constitute an argument against the tarring of roads, for, when tarring is well done, it diminishes the chance of injury to the eyes.

In 1911, Mirande[4] investigated the many substances prepared from coal-tar used for preserving wood, destroying moss on tree trunks, as insecticides, etc. He was led to conclude that these all contained creosote and gave off vapors which were destructive to leaves, flowers, shoots, etc., in the same way as coal-tar vapors, blackening them and causing death by plasmolysis. More recently the statement that the use of tar on roads has an injurious effect on the surrounding trees and vegetation, for which Mirande is held responsible, has been pronounced to be unfounded by German experts. Among others, H. F. Fischer studied the matter, and he presented some results before the International Road Congress which convened in London in

[1] *Compt. rend.*, **151**, 593.
[2] *Compt. rend.*, **151**, 769.
[3] *N. Y. Med. J.*, Nov. 30, **1912.**
[4] *Compt. rend.*, **152**, 204.

June, 1913. It may also be noted that the conclusions of Mirande respecting tarred roads have been refuted by several French engineers,[1] who have indicated that a large street in Bordeaux was treated with tar for some years without the slightest damage to the trees bordering the street, and that other towns also practised tarring without injurious results, though one example is on record where the trees around a square were destroyed by tar. In this case—at Fontenoy de Comte—the tar was spread so close to the trees that it prevented the water from getting to the roots.

[1] *Chem. World*, **2**, 106.

## CHAPTER XVI

## SOME PROBLEMS OF THE PETROLEUM INDUSTRY

While it is tacitly accepted by modern chemical industrialists that their manufacturing operations must be based upon research if they are to meet with success,[1] a number of manufacturers are even now certain that research will not pay. Some regard their technology as an hereditary art. Others have favorable raw material conditions or a large demand for their products, and are therefore disinclined to invest a very small portion of their earnings in a reserve of knowledge. Still others have prospered because of high tariff, notwithstanding short-sighted management. But most of our industries are built upon stronger foundations. It is plain that the use of natural laws offers a more stable basis upon which to erect a manufacture and a more uniform source of profit than any structure built upon artificial conditions created by pure empiricism or legislation. Moreover, the quality and value of a product are based upon the application of correct principles in its conception, preparation and use, and these correct principles can only result from scientific research. Ample support to this contention is to be found in the manufacturing operations of to-day.[2]

Consequently the industrial researcher, who deals with the processes of manufacture and the phenomena of reactions involved, is becoming less and less regarded as a burden unwarranted by returns. The aim of every industrial operation is toward perfection, both in process and the necessary mechanical equipment, and every new development in manufacturing creates new problems. It follows, then, that the greater the number of

[1] On the value of research to industry, see the senior author's address in *Science, N.S.*, **40** (1914), 871 to 881. The junior author has discussed the methods employed in the attack of industrial problems in *Sci. Mon.*, **1** (1915), 86–92. On what the scientific investigator has done and can do for industry, see the address by the senior author in *Sci. Am. Suppl.*, **80** (1915), No. 2081, 334–6.

[2] For a convincing demonstration, see HESSE, *J. Ind. Eng. Chem.*, **7** (1915), 294.

researches, the greater is the progress in a given field, and the greater becomes the number of new problems. Moreover, one can only conclude that, since perfection is but, after all, an ideal, no industrial field has been sufficiently investigated.[1]

The thirst for distinction and wealth kindles the lamp of invention, and the light of the knowledge resulting from discoveries and improvements in manufacturing operations has so emboldened us that some industries now consider themselves capable of solving any problem. This has been shown in innumerable instances, but is particularly true of the great petroleum industry, which, while its achievements have been stupendous, is nevertheless confronted with many problems of importance—problems which petroleum technologists are capable of clearing up, provided the service of research is called to their aid, but which have so far remained partially or completely unsolved.

Certain petroleum industrialists have frequently availed themselves of the service of research, to assist both in the perfecting of processes and the necessary mechanical equipment, and marked success has attended scientific progress in the refining of petroleum. In fact, some of the largest refiners now concede that chemistry is the intelligence department of the petroleum industry and gratefully acknowledge that the efficiency of their plants has resulted largely from research. Those companies which stand well to the fore maintain one or more research laboratories, and industrial research has left its mark on their development. It is, however, essential, particularly for the future welfare of the industry, that there be an increment in research activity; this may be brought about in a measure by the organizations represented in the petroleum industry and by our highly efficient Bureau of Mines, but there must be greater coöperation between petroleum industrialists and the universities and scientific societies, if the desirable fruitful research is to be stimulated.

Principally owing to the lack of reliable general information, the petroleum industry is, in many ways, groping in the dark. The same errors are repeatedly made in the same territory by different operators, and in both drilling and refining the various factors having a known influence are imperfectly understood in their relation one to another. Geology, the real foundation

[1] On the problems of chemical industry, see the senior author's paper in *idem*, 7 (1915), 535.

of a study of production, is but little applied or understood by the average producer, and in consequence much of the production is accidental rather than scientific.[1] When a producer finds oil he is simply a producer, and not a marketer, for he has usually no knowledge of the value of his product other than the posted price. He knows only in a rather vague way what characteristics are valuable, and when he has sold his crude he is frequently no longer concerned with the petroleum industry. The refiner usually has only the knowledge gained by an apprenticeship with an older refiner, and is therefore unfamiliar with the chemistry of refining.

Petroleum marketers maintain that the greatest need of those interested in the petroleum industry to-day is uniformity in methods of testing and in the physical instruments employed therein. Constant progress is being made in the analytical chemistry of petroleum and its products, but much research will be necessary before procedures are worked out which will be satisfactory for general adoption by the state inspectors and by the petroleum associations.[2]

From the industrialist's viewpoint, the great problem in the petroleum industry is flexibility in refining, in order that only such products as are in demand may be produced. To illustrate, let us assume that a certain petroleum yields, on refining, 30 gal. of gasoline, 20 gal. of turpentine substitute, 15 gal. of illuminating oil, 15 gal. of gas oil, 10 gal. of viscous neutral oil, and 6 gal. of steam-refined cylinder stock.[3] It is desirable that a refining process be devised which will enable that refiner to produce only the products for which there is a big market—at present, gasoline and lubricating oils.

Some technologists look forward to the time when the desired finished products, and only these, will be obtained from petroleums by purely physical methods, thus dispensing with chemical treatment.

Recently petroleum research has been centered on working out processes for the dehydration[4] and desulphurization[5] of

[1] See Chap. IX. On problems in connection with production, see Chap. X.

[2] See pp. 124, 127, and 517.

[3] See p. 497.

[4] See p. 528.

[5] See p. 609.

crude oils and for the depolymerization[1] of heavy oils (particularly the conversion of gas oils into motor fuel). Much success has resulted from these investigations, but the ideal processes remain to be found, especially those for converting products of the destructive distillation of residuum or heavy oils into saturated hydrocarbons. If hydrogenation could be successfully conducted on a commercial scale, the customary treatment with sulphuric acid could be dispensed with. Hydrogenation with platinum black or nickel could be accomplished if an antidote were known for the poisonous effect of sulphur compounds on the catalyst. Another matter of interest here is the polymerization of olefines; this subject requires broad study.

Automobile engineers should have available exhaustive data regarding the specification and use of petroleum products for internal-combustion and lubrication purposes. So far little has been accomplished in devising ways and means to use the lower grades of gasoline in internal-combustion engines; no exact work has been done on the efficiency of mixtures of casing-head gasoline with heavier distillates as compared with straight cuts; and, while it is known that gravity is not indicative of the value of a light distillate,[2] sufficient data has not been accumulated to educate the public to a rational basis of sale. Lubricating oils for special purposes have not received due scientific attention, and, as a result, the selection of a lubricant is more frequently casual than scientific.

Illuminating oils have been used and are being used more and more in motors of heavy trucks. In order to use these distillates in the ordinary motor, it is only necessary to heat the carburetor by means of the exhaust gases. The motor must be started with gasoline or alcohol; but it is easy to attach a small supplementary tank.[3] The great drawback to the use of illuminating oils, or kerosenes, is their production of polymerized products in the cylinder, and a large number of mixtures have been proposed to obviate this difficulty. It is said[4] that kerosene and

[1] See p. 554.

[2] The specific gravity is not a sufficient indication of the suitability of a sample of gasoline for any given purpose, since the chemical composition of samples of the same specific gravity is different for oil of different origin.

[3] On kerosene as a motor fuel, see MERSEREAU, *J. Soc. Chem. Ind.*, **33** (1914), 626.

[4] SELDEN, *Petroleum*, **9** (1914), 1304; *Nat. Petrol. News*, **5**, No. 7, 39; *Petrol. Rev.*, **29**, 463.

other heavy petroleum oils can be utilized as fuel for internal-combustion engines if small quantities of peroxides or other highly oxygenated substances, such as picric acid, naphthalene picrate, or similar nitrated compounds of naphthalene,[1] phenanthrene, pyrene and picene, are either dissolved in them or are sprayed into the air inlet of the carburetor. Perhaps "peroxidized kerosene" may furnish the solution of this problem, although automobile engineers are hopeful that there will be devised a gasifying apparatus which will give no trouble from coking or partial combustion, and progress has, in fact, been made in this direction.

The problem of the better utilization of illuminating oil may be eventually solved by processes for converting it into products for which there is a demand, as, for example, acetylene.[2]

In the chemical treatment of petroleum products, systematic research with the view of ascertaining the most favorable temperature for the acid treatment of petroleum distillates, would be of great practical value as affording an indication to the refiner how to proceed under all circumstances.[3] A well-known problem in refining is the chemical treatment of lubricating oils in such a manner as to minimize the losses and to prevent the emulsions which result when sulphuric acid is used. The production of persistent emulsions in the refining of petroleum is a problem to be attacked from the colloid-chemical point of view. An economic process is wanted for isolating pure naphthenic acids of high molecular weight from waste lyes.[4] Another problem is the utilization of the esters from the naphthene carbo-acids[5] contained in the residue from the alkali treatment. A question of interest here is, what is the cause of the malodor of naphthenic acids,[6] and how may these products be most effectively and economically deodorized?[7]

The following are some of the many other problems con-

[1] On the employment of nitronaphthalene for this purpose, see DE COSMO and QUINAUX, French Patent 459857, Apr. 10, **1913.**

[2] On the production of acetylene from various organic substances, see TRUCHOT, *Compt. rend.*, **84,** 714; and DESTREM, *idem*, **99,** 138.

[3] Cf. p. 583.

[4] PYHÄLÄ, *Chem. Rev. Fett- Harz- Ind.*, **21** (1914), 128.

[5] On these acids, see FUCHS and SCHIFF, *Chem.-Ztg.*, **19,** 1469.

[6] PYHÄLÄ (*Petroleum*, **9** (1914), 1506) attributes the odor to the presence of sulphur compounds, but this is not established.

[7] See SCHMITZ, *Mat. grasses*, **7** (1914), 4115, on the processes in use.

fronting petroleum technologists: A more rational utilization of the butanes and pentanes in natural gas gasoline and in light petroleum fractions; the separation of the vapors of light oils from steam vapors when petroleum is distilled by the introduction of superheated steam, and any steam not condensed with the oil vapors is returned to the superheater and passed again through the still; does the hot still metal exert any catalytic action in the pyrogenic decomposition of residues; the minimization of the decomposition of sulphur compounds during the distillation of petroleum containing asphalt; what rôle do sulphur compounds take in the condensation of petroleum to asphaltum; increasing the efficiency of tower stills; the rapid and effective revivification of spent fullers' earth;[1] the separation of the aromatic and cyclic unsaturated hydrocarbons from the paraffin and naphthene hydrocarbons by a more effective or cheaper reagent than those now in use; the fractionation of petroleum residues containing paraffin wax into distillate and undecomposed amorphous wax;[2] the elimination of gummy or asphaltic portions from wax distillate by a distillation or mechanical procedure; what constituents of a mineral oil are responsible for the lubricating power thereof,[3] and can the lubricating properties of an oil be increased in the light of this knowledge; the prevention of the oxidation of mineral oils in transformers;[4] products which will make emulsions with water without any oil coming to the surface; the recovery of more of the calorific value of an oil in the gas in producers, obtaining oil-gas of uniform quality during the whole of the gas-making period, and reducing the amount of the lampblack formed; and a rational plant for producing oil-gas from tar and of carbon monoxide from the

[1] On maintaining and improving the efficiency of fullers' earth, see M. J. WELSH, United States Patents 1132054 and 1159450 of **1915.**

[2] GRÄFE (*Petroleum*, **9** (1913), 303) states that the Kubierschky column answers for this purpose.

[3] MARCUSSON (*Mitt. kgl. materialprüf.*, **31** (1913), 301) has concluded that polynaphthenes are the main cause in the case of high-boiling oils; but this requires demonstration. Cf. MARCUSSON, *Chem.-Ztg.*, **35** (1911), 729 and 742.

[4] Transformers are made in which contact of the air with the hot oil is prevented; but these are only partially successful, since the air dissolved by the oil is able to produce deposits and acid formation (BRAUEN, *Chem. Rev. Fett- Harz- Ind.*, **21** (1914), 138 and 170).

residue, the mixed gases to be suitable for use in gas engines.[1] Then, too, notwithstanding the many processes which have been patented, methods for the solidification of petroleum and its products are still under investigation.

As mentioned in Chap. XII, petroleum is generally refined to obtain products which may be used as fuels or lubricants; but later, when there is evidence of a depletion of the national petroleum supplies and, owing to a waning production, petroleum will be unable to compete with, say, shale oil, it will be possible to obtain so many more valuable products from its distillates that the use of any one of these as a fuel will be condemned from the standpoint of conservation. Petroleum will, like natural gas, form the basis of a chemical products industry which will be as distinct as the coal-tar industry of today. Several of the largest refiners are preparing for the future by accumulating a reserve of knowledge through research.

Some attention is now being given to the production of drying or siccative oils[2] from petroleum. It is claimed by the De Bataafsche Petroleum Maatschappij[3] that an oil the drying properties of which are comparable with those of linseed oil, may be obtained by chlorinating a mineral oil fraction of the specific gravity 0.885 at 15°C., in such a manner that 1,000 liters of it absorb 800 kg. of chlorine, and then heating the chlorinated product with the addition of zinc dust to 290° to 300°C., until the chlorine has been quantitatively expelled as hydrochloric acid. The great difficulty is to produce an oil economically which will be devoid of tackiness.

Artificial resins may also be obtained by the chlorination of petroleum and its distillates.[4] The reduction of the time of the reaction and the purity of the products present the main difficulties.

One of the most important, as well as most difficult, problems is the synthetic preparation of fatty acids from petroleum.

[1] For a consideration of such a process, see DREXLER, *Oelmotor*, **2** (1913), 125, 309, 433 and 563.

[2] Oils having drying properties which make them suitable for use as varnishes or lacquers or as binding materials or vehicles for paints.

[3] English Patent 23376, Dec. 1, **1914**. For another process, see English Patent 2672, Feb. 1, **1913**, of the NEW OIL REFINING PROCESS CO., LTD.

[4] C. F. BOEHRINGER UND SÖHNE, German Patents 256856, Nov. 19, **1910**; and 258156, Dec. 28, **1911**.

While this problem has received much attention, there are still possibilities of solution.

Zelinsky[1] obtained good yields (up to 60 per cent.) of organic acids[2] by chlorinating different fractions of Baku petroleum, treating the products obtained with magnesium, subjecting the magnesium compounds to the action of carbon dioxide, and decomposing the complex substances formed with water and sulphuric acid. The production of fatty acids from liquid hydrocarbons is said to be commercially applicable by this process, but the cost of the product is still too high.[3]

When an economic process is available for the manufacture of fatty acids from petroleum, a working basis will thus be had for the production of edible products, for some of the acids obtainable may be converted into glycerides.

For the manufacture of isoprene, the lowest boiling fraction of petroleum, of the composition $C_5H_{12}$, may be used: by systematic chlorination, isomerization, and liberation and addition of hydrochloric acid, the three hydrocarbons present (iso- and normal pentane and tetramethylmethane) can all be converted into isoprene.[6]

It may be predicted that a simple, direct process for obtaining the butadiene hydrocarbons from petroleum will soon be found, and thus a good route to synthetic rubber will be opened up. Pyhälä[5] has shown that the fraction of Baku petroleum boiling between 98° and 106°C. yields up to about 20 per cent. of its weight of adipic acid, which, by way of the amide, can be converted into butadiene. This method of preparation is, however, hardly possible from a commercial standpoint, and butadiene is usually obtained from benzene or phenol.[6]

It is of interest to mention here that the emulsions obtained with the turpentine-like and other hydrocarbons of petroleum

[1] *Z. angew. Chem.*, **16** (1903), 37.

[2] Acids of the sebacic and hydro-aromatic series (French Patent 326665, Nov. 25, **1902**).

[3] Colletas, *Mat. Grasses*, **7** (1914), 4151. On the formation of dibasic fatty acids of the oxalic acid series by the oxidation of Baku petroleum fractions with nitric acid, see Herr, *Petroleum*, **5** (1910), 692.

[4] See French Patents 435312, Oct. 14, **1911**; and 452246, Dec. 20, **1912**, of the Badische Anilin und Soda Fabrik.

[5] *Neftjanoje Djelo*, **1913**, No. 7; *Petroleum*, **9** (1914), 1376.

[6] For a review of recent work on synthetic rubber, see Holt, *Z. angew. Chem.*, **27** (1914), 153.

by the aid of certain emulsifying agents, are similar to rubber latex and can be worked up into rubber-like products.

Finally, it may be noted that the production of dyestuffs from petroleum is an open field for research.[1] It is said[2] that the nitro-derivatives obtained by treating machine or spindle oils with warm nitric acid (specific gravity, 1.50) are closely related to the nitro-compounds obtained by nitrating polynaphthenic acids and used in the preparation of dyestuffs. Nitro-products which yield with alkalies brown substantive dyestuffs and with sulphur and alkalies brown to brown-violet sulphur dyestuffs, have been obtained by the nitration of the higher fractions from Galician petroleum.[3] The preparation of aromatic hydrocarbons from fatty hydrocarbons—especially the preparation of benzene[4] and its homologues from petroleum—requires investigation along broad lines.

[1] On a brown dyestuff from petroleum, see FRASCH, English Patent 10320, May 25, 1893; and on a yellow dyestuff, see FRASCH, English Patent 10323, May 25, 1893. See also page 865.

[2] KHARITCHKOV, *Chem.-Ztg.*, 37 (1913), 869.

[3] FREUND, *Z. angew. Chem.*, 25, 1058.

[4] For patented processes, see, among others, HAUSMANN and PILAT, German Patent 227178, Apr. 1, 1909; and HOLCGREBER, French Patent 460827, July 28, 1913. See pp. 570, 576 and 579.

## CHAPTER XVII

## THE SHALE-OIL INDUSTRY

In the United States oil-shales[1] occur in Kentucky; over large areas in the middle portion of the Green River formation in northwestern Colorado and northeastern Utah; on the Humboldt River, opposite to Elko, Nev.; on the Big Blackfoot River and near Great Falls, Mont.; and in the Cholame Valley, north of Parkfield, Cal. Some of these deposits are of good quality and may be said to constitute a fuel as well as a fertilizer reserve of decided economic possibilities.[2] While these resources may not be utilized until there is convincing evidence of the depletion of the national petroleum, and possibly coal,[3] supplies, their availability and latent possibilities have aroused interest,[4] and there has been some discussion of how they might be worked with greatest profit.[5] This chapter has been prepared, in anticipa-

[1] Shales which contain an *oil-forming* substance, "kerogen," or bituminous matter which gives rise to oily and tarry matters upon destructive distillation. Reference is not had here to *oil-bearing* shales, from which petroleum may be obtained by mechanical means.

[2] In this connection, see Baskerville, *Proc. Seventh Internat. Cong. Appl. Chem.*, **1910**, Sect. IV., Al, 22; *Eng. Min. J.*, **88**, 149 and 195; Baskerville and Hamor, *J. Ind. Eng. Chem.*, **1** (1909), No. 8, 507; and *Orig. Com. Eighth Internat. Cong. Appl. Chem.*, **25**, 631.

[3] As pointed out in Chap. V, various bituminous materials were distilled for the production of illuminating oil by fifty-three "coal oil" companies about 1860; but the process followed was very crude and ammonium sulphate recovery was never attempted. Consequently, although many of the coals and other materials used were of high grade, and merit investigation as to yield of oil, gas, and ammonium sulphate according to modern practice, it is not surprising that the refining of petroleum paralyzed the industry. See p. 203.

[4] See Day in "Mineral Resources of the United States," **1913**, ii, 1071; Woodruff and Day, *Bull. U. S. Geol. Surv.*, No. **581-A**, 1–21; DeBeque, *Eng. Min. J.*, **99**, 773; *U. S. Comm. Rept.*, Mar. 13, **1916**; and Winchester, *Bull. U. S. Geol. Surv.*, No. **641-F**.

[5] By Ells and Hamor, *Canada Dept. Mines, Report* No. **55**, *Mines Branch*, **1910**; Baskerville and Hamor, N. Y. Sect., Soc. Chem. Ind., Apr. 23, **1909**; and Baskerville, Forty-ninth Meeting, American Chemical Society, Apr. 8, **1914**. Reference should be made to the report by Ells and Hamor for information regarding the oil-shales of Canada.

tion of future investigations, along broad lines, of American oil-shales, in order to indicate, mainly from Scottish experience, the principal factors involved in shale-oil technology.

## THE BEGINNINGS OF THE SHALE-OIL INDUSTRY

In 1694, Eele, Hancock and Portlock made "pitch, tar and oyle out of a kind of stone from Shropshire," England;[1] and in 1681 Becher and Searle took out a patent for making pitch and tar from pit coal.[2] Clayton, however, made the first investigation of the spirit produced by the distillation of coal.[3] As early as 1761, oils were distilled from black bituminous shales for medicinal purposes,[4] and it was found that they could be used instead of both oil of turpentine and "oil of petre."

The Earl of Dundonald obtained oil from coal by destructive distillation in 1781, and he later procured a patent for manufacturing various products from coal.[5] About 1812, it was known that an oil resembling turpentine could be made from coal-tar by destructive distillation,[6] as well as red and black varnishes;[7] and in 1815, a works near Sunderland, England, belonging to Messrs. Featherstone, produced "petroleum" and ammonia from coal.[8]

In 1830, Laurent obtained paraffin by the distillation of bituminous shale, and three years later he suggested working the Autun shale in Saône-et-Loire, France.[9] Selligue manufactured products from this shale, and secured patents[10] for the manufacture of paraffin on a commercial basis; his process for distilling bituminous schists was considerably developed before 1845, and it is of interest to note that ammonium sulphate was manu-

[1] English Patent 330, **1694**; Eele, *Phil. Trans.*, **19** (1697), No. 228, 544; *Phil. Trans. Abridged*, **4**, 168.

[2] BECHER'S "Fossil Fuel," **1683**, 404.

[3] *Phil. Trans.*, **41** (1739), 59.

[4] LEWIS' "Materia Medica."

[5] "Description of the Estate and Abbey of Culross," 34; WILLIAMS' "Mineral Kingdom," **1789**, **1**, 253.

[6] HODGSON'S "Account of the Felling Explosion," **1813**, 25.

[7] SURTEE'S "Durham," **1820**, **2**, 85.

[8] HOLMES' "Treatise on the Coal Mines," **1816**, 18.

[9] *Ann. chim. phys.*, (2), **54** (1833), 392; *Compt. rend.*, **4** (1837), 909.

[10] French Patent 9467, Nov. 14, **1838**; addition, Mar. 27, **1839**; English Patent 10726, **1845**. The latter patent was taken out under the name of DE BUISSON. See also SELLIGUE, *Compt. rend.*, **4** (1837), 969; **9** (1839), 140; and **10** (1840), 861.

factured. Shale-oil is still produced in France, and five companies are working Autun and Buxière-les-Mines bituminous shales. Four out of six works in France have been constructed on the Scotch system, and normally about one-tenth as much shale is retorted in France as in Scotland. The shale now mined in France resembles Boghead coal and yields about 50 gal. of crude oil per ton.

## THE SCOTTISH SHALE-OIL INDUSTRY[1]

James Young, of Kelly, Scotland, was the first to produce oil from bituminous minerals on a commercial scale in Scotland, and it is to him that the shale-oil industry in its present form owes its existence. In December, 1847, Young learned of the Alfreton, Derbyshire, petroleum, and, in company with Meldrum, started work in 1848 manufacturing oils from this material. Upon the exhaustion of the supply of petroleum, Young attempted to imitate the natural processes by which he thought the oil had been formed. He learned that an oil containing paraffin could be produced from bituminous coals by slow distillation, and made this "invention" the subject of a patent.[2] In 1850, Young, Meldrum and Binney erected a plant at Bathgate for the distillation of Boghead coal, and crude oil was produced at this plant in 1851. About this time, works were established in Germany for producing paraffin oil from shale, the most important being the plant of A. Wiesmann & Co., at Augustenhutte, near Bonn.

In 1851, two oil works were in operation in Scotland, the Bathgate Works and the Crofthead Oil Works; but in 1852 the latter works was closed. In 1857, three oil works were in existence, and this number was increased to 19 in 1862, the year in which the Torbanehill mineral[3] became exhausted, and gas and parrot coals and oil-shale were resorted to. While torbanite yielded from 96 to 130 gal. of crude oil per ton, the shales first used furnished only 45 gal.

Young's patent expired in 1864, in which year 38 new works were erected; but about this time the industry received a severe check on account of the importation of kerosene into the British Isles from the United States. In addition, competition had

[1] ELLS and HAMOR, *loc. cit.;* BASKERVILLE, *Eng. Min. J.*, **88**, 149.

[2] English Patent 13292, **1850**; see also YOUNG, *Proc. Scot. Soc. Arts*, **7** (1868), 36.

[3] See p. 917 for an account of Torbanehill mineral, or torbanite.

caused prices to fall greatly. The price of burning oil was 2s. 6d. per gallon from 1850 to 1864, and 1s. 6d. soon after the expiration of Young's patent. In 1871, 51 works were in operation in Scotland; in 1880, only 19 were running, and in 1895 but 12.

At the present time the number of companies refining shale oil in Scotland is four; these are as follows: Young's Paraffin Light and Mineral Oil Company, Ltd., which was organized in 1866, and has crude oil works at Addiewell, Bathgate and Uphall; the Oakbank Oil Company, Ltd., which was registered in 1886 to take over the properties of a company of the same name, established in 1869; the Broxburn Oil Company, Ltd., which was started in 1877, and has crude oil works at Broxburn and Roman Camp; and the Pumpherston Oil Company, Ltd., which was formed in 1883, and has crude oil works at Pumpherston, Tarbrax, Seafield, and Deans.

The development of the Scottish shale-oil industry has been carried out with skill and resource, and it is to be regretted that the industry has not met with the entire commercial success it well deserves. Ever since 1850, it has only been by extremely careful management and the constant and intelligent application of science to the improvement of processes and to the utilization of waste products, that the oil refiners of Scotland have been able to hold their own. However, the success of the companies now in operation is shown by the dividends declared annually.

Following the granting of a patent to Young for a process of producing paraffin from bituminous minerals, various companies were organized in Flintshire, England;[1] but after the initial development of the Pennsylvania oil fields, retorts costing $300 were sold for $15 to scrap-iron dealers.

## THE OIL-SHALES OF SCOTLAND

The Scotch oil-shales are a peculiar group of rocks which occur in a somewhat broad and well-defined belt, lying largely to the west of Edinburgh, but are developed on both sides of the Firth of Forth. They are described in the *Memoirs of the Geological*

[1] These companies, which were all of small proportions, were as follows: Robottom & Co., Ferney Company, Flintshire Oil Company, Williams Oil Company, Coppa Colliery Company, Coppa Oil Company, and Padeswood Oil Company. About this time, a company, the Bituminous Shale Company, was organized in Dorsetshire, and a plant was erected in Weymouth. In 1854, the works were removed to Wareham, and the company dissolved in 1872. An inferior cannel coal was distilled in Wales about the same time.

*Survey of Scotland* under the name of the Calciferous Sandstone series. Oil-shale[1] is a fine-grained, brownish (sometimes brownish black and black) clay shale, which can be readily recognized in the field. It has a laminated or horny fracture and possesses a specific gravity of about 1.75. A true oil-shale does not coke when heated to redness in a closed vessel.

The brownish oil-bands are interstratified with non-bituminous or less bituminous beds, sometimes of a bluish gray color, known to the miners as "blaes;" and when destitute of bituminous matter and occurring as barren blaes, or ribs of hard calcareous or quartzose matter, as "kingle." The blaes variety decomposes much more readily on exposure to the weather than the oil-shales; and the bluish gray color (whence the name blaes), when the shale is decomposed into clay, is easily noted. The oil-shales, from the presence of the bituminous matter, strongly resist the action of the atmosphere and remain for years on the dump, the brown shale of the surface changing to a bluish gray tint. The beds or bands are persistent for long distances, but sometimes lose their bituminous character, and pass into the bluish or blaes variety.

The oil-shales of the district are known as plain and curly, the plain being flat-surfaced, often with a slickensided aspect, due doubtless to sliding, through the agency of faults, while the curly variety is somewhat contorted or curled. They are both quite soft and free from grit, yielding readily to a sharp knife, and can be cut off in thin shavings without breaking during the process of cutting.

The value of Scotch oil-shale, as now mined, does not entirely depend upon the yield of oil by distillation, and, as a rule, those now worked do not seem to be as rich in kerogen as in the earlier days of the industry. Much of the present value depends upon the percentage of nitrogen and consequently upon the yield of ammonia during retorting. The amount of crude oil now obtained by retorting Scotch shale rarely exceeds 30 gal. per ton, and shales yielding as low as 10 to 15 gal. are being retorted, owing to the fact that the yield of ammonia is sometimes larger in shales poor in oil yield than from those of high kerogen content. The yield of ammonium sulphate is, therefore, a most important factor in estimating the profits arising from the distillation of shales, because of the extensive demand for this product. In

[1] Also known as pyroschist or bituminous shale.

operating shale deposits, the matter of by-products must always be carefully considered.

**Scottish Oil-Shale Seams.**—The highest seam formerly worked for oil in the West Lothian district was the Torbanehill mineral, already referred to. This occurred at the base of the coal measures, overlying the Millstone-grit. In this connection it holds the same geological position as the stellarite found in the Pictou coal-basin of Nova Scotia, where this mineral occurs near the base of the coal measures and overlying the Millstone-grit of that area. In the list of seams which occur in the Scotch shale field, this bed of Torbanehill mineral may be styled No. 1 as regards position.

The shales worked from time to time in the Scotch areas, several of which have long since been abandoned, may be described in the following descending order:

No. 2.—A seam of 11 in., Levenseat shale, yielding 29 gal. of crude oil per ton; worked probably as the parrot coal seam; belongs to the Lower Carboniferous limestone formation.

No. 3.—From the Calciferous Sandstone formation. The upper shale is the Raeburn or Damhead shale, 3 ft. to 5 ft. thick, sometimes 6 ft., including layers of blaes or fireclay. Worked at West Calder, Tarbrax, and Charlesfield. The yield of oil was 54½ gal.; ammonium sulphate, 14 lb.

No. 4.—Mungle or Steuart shale, 2 ft. thick at West Calder. A 2-ft. seam of coal was worked at Broxburn by Robert Bell, together with 17 in. shale, yielding 26 gal. of oil per ton. There were 4 in. of ironstone above the shale. At another point in the Broxburn field there were 17 in. of shale, giving 41 gal. of crude oil per ton, the average yield being 34 to 35 gal. oil and 30 lb. ammonium sulphate.

No. 5.—The "grey shale" of Addiewell, about 20 in. thick. It gave crude oil, 28 gal.; and ammonium sulphate, from 13 to 18 lb.

No. 6.—The Fells shale, from 3 ft. to 5 ft. thick at West Calder, and in places about 7 ft.; mined at Addiewell, Tarbrax, Breich, Seafield, Hermand, and Pentland. Yield of oil, from 26 to 40 gal.; of ammonium sulphate, 20 to 35 lb.

No. 7.—Wee shale of Oakbank; 2 ft. thick; not worked; oil, 36 gal.

No. 8.—Big shale of Oakbank; 4 ft. 6 in. thick; formerly used at Oakbank; oil, 22 gal.

No. 9.[1]—Wild shale of Oakbank, or Lower Big shale, the gray shale of Broxburn; worked at Oakbank and Broxburn, 6 ft. thick; oil, 29½ gal.; ammonium sulphate, 34 to 41 lb.

No. 10.[1]—The Curly shale of Oakbank and Broxburn; mined at Broxburn, Oakbank, and Dalmeny; 6 ft. thick; oil, 22 gal.; ammonium sulphate, 35 lb.

No. 11.[1]—The Broxburn shale, which varies in oil from 10 gal. to 50 gal. Includes several seams, of which at Broxburn there are three: the Broxburn Gray, 6 ft. thick, giving about 23 gal. of oil and 35 lb. of ammonium sulphate; the Broxburn Curly, 5 ft. 6 in. thick, with 26 gal. of oil and 38 lb. of ammonium sulphate; and the Broxburn, from 5 to 6 ft. thick, giving oil from 28 to 35 gal., and 40 lb. of ammonium sulphate.

No. 12.[1]—Lower Wild shale of Oakbank, 5 ft. 6 in. thick. It gives 19¼ gal. of crude oil per ton.

No. 13.—The Dunnet shale, from 4 ft. to 12 ft. thick. This yields 24 to 33 gal. of oil and 24 lb. of ammonium sulphate. It varies considerably at different points.

No. 14.[1]—The Oakbank New shale, 8 ft. 6 in. thick. It gives 20½ gal. of oil.

No. 15.[1]—The Barracks shale, sometimes 8 ft. thick. It yields from 18 to 22 gal. of oil.

No. 16.—The Pumpherston seams, five in number, as under:

1.[1] Jubilee seam; thickness, 8 ft.; oil, 18 gal. ammonium sulphate, 55 lb.

2. Maybrick seam; 5 ft. thick; oil, 17 gal.; ammonium sulphate, 60 lb.

3.[1] Curly seam; 6 ft. thick; oil, 20 gal.; ammonium sulphate, 60 lb.

4.[1] Plain seam; 7 ft. thick; oil, 20 gal.; ammonium sulphate, 60 lb.

5.[1] Wee seam; 4 ft. thick; oil, 18 gal.; ammonium sulphate, 60 lb.

Regarding the thickness of the oil-shales worked in the Scotch field, this varies greatly in different parts, or even in different portions, of the same mining area. The oil-bands sometimes increase very materially, and vary from a few feet or even inches in thickness, to 6, 8, 10, and in some cases 15 ft., interstratified with blaes or bluish gray portions less rich in bituminous matter, or with hard calcareous or silicious bands.

[1] Seams now worked.

It is practically impossible to obtain, in any portion of the Scotch fields, continuous sections of the shale beds at the surface, owing to a widespread mantle of drift, and the consequent paucity of good sectional exposures. In consequence, the geological structures of the several districts have been largely obtained by a close study of the underground workings and from the numerous bore-holes. Certain bands of rock thus struck are easily recognized, and serve as fixed points to determine positions.[1]

**The Mining of Oil-Shale.**—Shale mining is carried on very much in the same manner as the mining of bituminous coal. The roof has to be supported with the usual timber props, provision made for pillars, and a regular system of roads, driveways and drifts installed. In Scotland, powder in small charges is generally used as an explosive, owing to the soft nature of the oil-shale.

In the regular process of mining, the broken down shales are brought direct to the surface by wire haulage, and transferred thence to the breaker, into which the loads are discharged prior to the commencement of the retorting.

While it varies according to conditions, the cost of mining shale may be placed at 5s. per ton. Miners' wages are fixed periodically, and are about as follows: Miners . . .6s. 3d. per day; helpers . . .5s. 9d. per day. Two men usually work together, and produce jointly about 8 tons per day on a seam 6 ft. to 7 ft. thick, or an average of 4 tons per man; and on a smaller seam, say of 5 ft. to 6 ft., about 6 tons for each shift of two men. 3,268,666 long tons of oil-shale were produced in Scotland in 1914.

### THE TECHNOLOGY OF THE SCOTTISH SHALE-OIL INDUSTRY

**The Crude Oil Works.**—The crude oil works consists of retorts, arranged in benches, the necessary condensers and scrubbers, the naphtha recovery plant, and the ammonium sulphate plant. The shale as it is received at the mine head is transferred to the retort department of the works by means of wagons, which are emptied into the shale-breakers by hydraulic machinery, and there the shale is broken into small pieces, about 6 in. square, by

[1] On the geology of the Scottish oil-shales, see CADELL and GRANT-WILSON, *Mem. Geol. Surv. Scotland*, **1906**; and CADELL, *J. Geol.*, **2** (1894), 234; *Trans. Edinb. Geol. Soc.*, **8** (1901), 116; and *Trans. Inst. Min. Eng.*, **22** (1902), 314.

passing it between two toothed drums or rolls, about 9 ft. long, which are driven by gearing from a steam engine. The breakers



FIG. 303.—Shale-breakers of the type in use at Broxburn, Scotland.



FIG. 304.—The discharge from the bottom of a shale-breaker, showing the pair of toothed rolls.

are made of cast-iron discs, about 3 ft. in diameter, and are provided with heavy teeth on their periphery; the tooth clearance is about 1 in. Each breaker crushes about 200 tons of shale per

day of 8 hr., and there is one or more of them, according to the amount of shale required (Figs. 303 and 304). From the breaker, the shale drops into iron hutches, holding from 10 to 25 cwt. each, which are hauled up an incline to the top of the retort bench. The hutches are raised by an endless chain (Figs. 305 and 306), set in motion by wheels geared from the engine which runs the breakers, and are so constructed that they are easily



FIG. 305.—Conveying oil-shale from the breakers to the retorts, Broxburn, Scotland.

tipped for discharging into the top of the retorts. Four laborers are required for each breaker.

The crude oil works is generally located in the center of a shale field, and in this way considerable expense is saved in haulage.

*Retorts.*—As noted, oil-shales (bituminous shales) and coals do not contain free oil or oily matter—that is, they are not oil-bearing—but must be subjected to destructive distillation at low temperatures, in order to obtain a crude oil from them. This

distillation is carried out in retorts, of which many forms have been devised (see Figs. 307 and 308). At the present time, how-

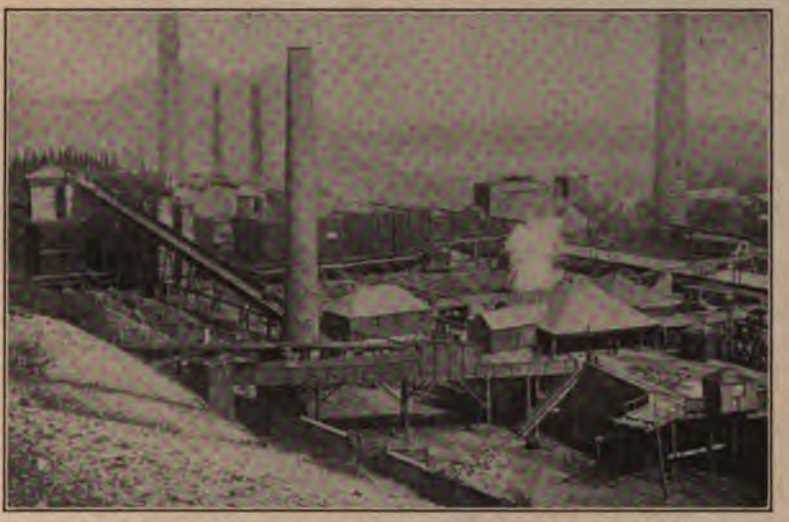


FIG. 306.—A general view of the refinery of the Pumpherston Oil Company, Ltd., showing the endless-chain haulage-incline to the top of the retorts.

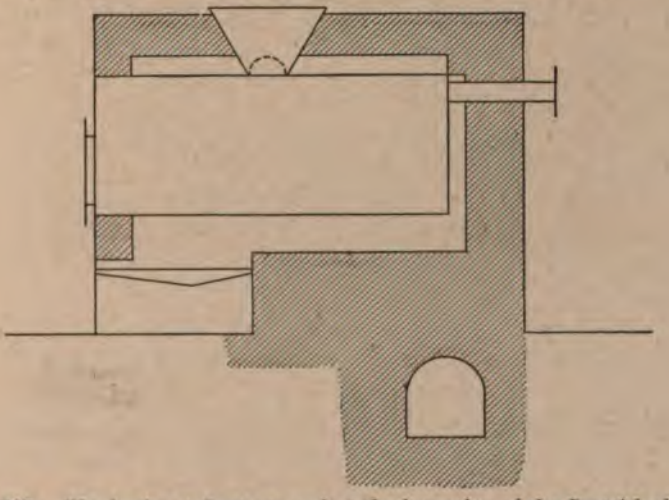


FIG. 307.—The horizontal retort used at the inception of the Scottish shale-oil industry.

ever, there are three types of retorts in operation, viz., the Pumpherston, the Henderson, and the Young and Fyfe. All of these retorts are based on the Young and Beilby principle.

The Young and Beilby retort (Fig. 309), which was patented in 1881,[1] consisted of four iron retorts, connected with a common hopper above. Each retort was provided with an iron upper part, in which the oil was distilled from the shale at the "lowest possible temperature" (about 900°F.); after the oil had practically all distilled, the shale passed down through the lower portion of the retort, which was built of firebrick. In this section of the retort the dekerogenated shale was subjected to a high temperature (about 1,300°F.), in the presence of steam,

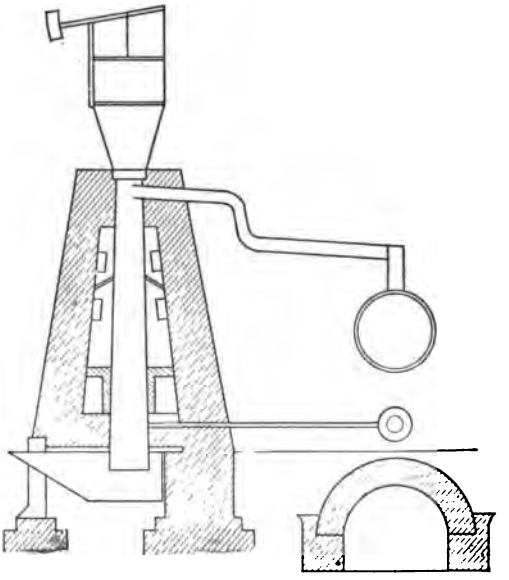


FIG. 308.—The vertical retort used in the early days of the Scottish shale-oil industry.

thereby converting the carbon into a mixture of carbon dioxide and carbon monoxide, and the nitrogen into ammonia. About 1 lb. of steam per square foot of shale was used, and the shale remained in the retort for 18 hr. Every 6 hr. about 8 cwt. of broken shale were put in the top hopper (jumbo), and at the same time the spent shale was removed from the bottom hopper. A bench of 80 Young and Beilby retorts had a throughput of 100 to 120 tons of shale per 24 hr. In 1894, the quantity of shale distilled in retorts of this type amounted to 92 per cent. of the

[1] English Patent 4284.

production of Scotland. The cost of retorting 1 ton of shale in retorts of this principle was 24d.

The Pumpherston, or Bryson, retort[1] (Fig. 310) was devised by Messrs. Bryson, Jones, and Fraser, and is used by the Pumpherston and Oakbank companies with success. It has been in use on an extensive scale since 1896, and there are now about 1,500 retorts on this principle in operation in Scotland. The experimental retort erected by James Bryson at the Pumpherston

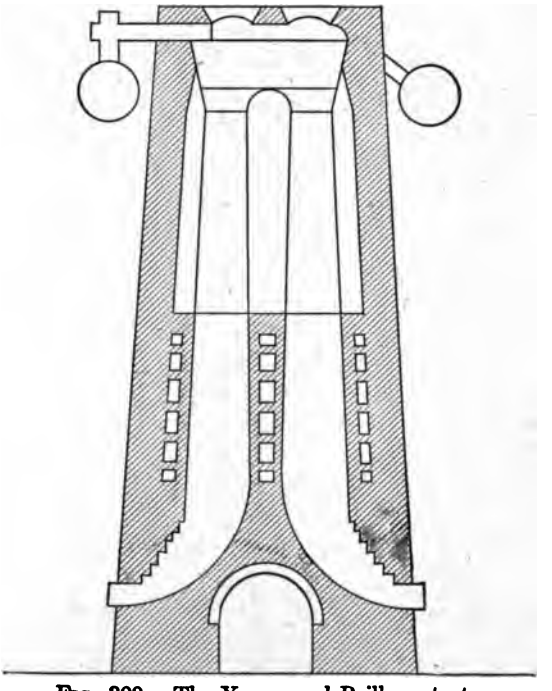


FIG. 309.—The Young and Beilby retort.

works, in 1894, was the first constructed on this plan. It was found that, in addition to reducing the cost of working by one-half, as well as improving the yields got from the shale, it obviated the difficulties and heavy losses caused by the shale fluxing in the older types of retorts, which were only moved intermittently, the continuous moving of the whole mass of shale in the new type preventing the fluxing, reducing the up-keep from a heavy charge to a nominal figure, and prolonging the life of the retort

[1] English Patent 7113, 1895.

to an extent as yet undetermined, as the first bench built, in 1896, is still being worked.

The Pumpherston retort consists of two portions: the upper or cast-iron portion, which is 15 ft. in length, 2 ft. in diameter at the top, and 2 ft. 4 in. in diameter at the bottom; and the lower or firebrick portion, which is 20 ft. in length, 2 ft. 4 in. in diameter at the top, where it joins the cast-iron portion, and 3 ft. in diameter at the bottom. At the end of the firebrick portion there is a disc support, or table, which supports the column of shale in the retort; this table is provided with a revolving arm, or quadrant, which removes the spent shale and maintains a movement in the shale column by revolving at regular intervals. At the top of the retort there are iron hoppers, holding 4 tons, 10 cwt. each, into which 1 ton of broken shale is charged every hour. The iron portion is heated to a dull red heat externally, and it is in this part of the retort that the destructive distillation takes place, the oil vapors passing out below the hoppers in an iron main, 2 ft. 6 in. in diameter. In the lower or firebrick part of the retort, the shale is subjected to a temperature sufficiently high to burn off all the carbon from the oil-spent shale; this is done in the presence of steam, and ammonia is produced by the hydrogen of the steam uniting with the nitrogen contained in the shale. About 60 per cent. of the total nitrogen is converted into ammonia and recovered. At the bottom of the retort there are hoppers, which converge in such a way that a single line of rails under the center of the bench of retorts re-

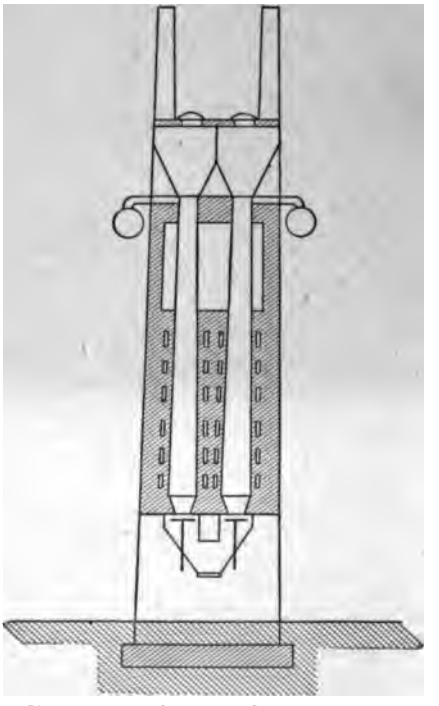


FIG. 310.—The Pumpherston retort.

ceives the spent shale, in an iron hutch, 5 ft. by 4 ft. 6 in. by 2 ft. (Figs. 311 and 312).

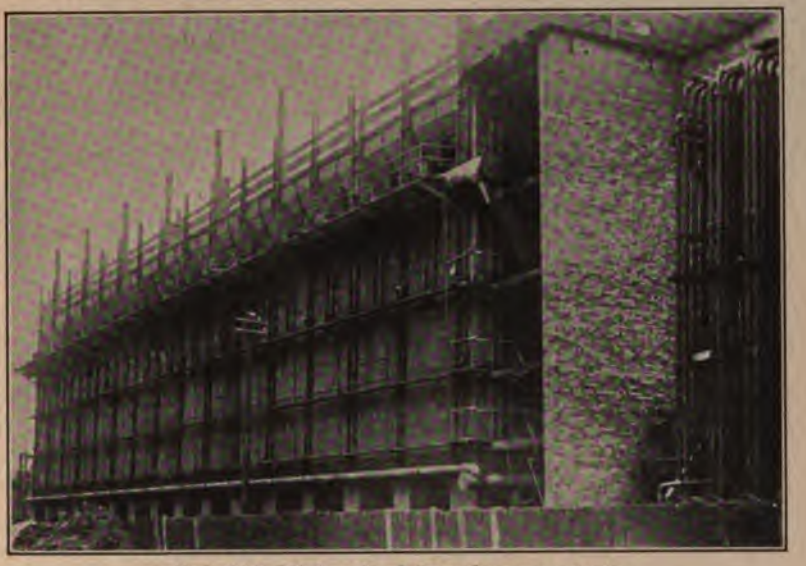


FIG. 311.—A bench of Pumpherston retorts.



FIG. 312.—Three benches of retorts at Pumpherston, Scotland.

In this type of retort, a throughput of 4 to 5 tons per day may be worked at a cost not exceeding 1s. 6d. per ton.

The cost of the Pumpherston retort, including condenser,

ammonia scrubber, and receiving tanks, but exclusive of the scaffold for charging, amounts to about £350.

The Henderson retort[1] (Fig. 313) is now used by the Broxburn Oil Company, Ltd., in an improved form.[2] This type of retort is 60 ft. high from the ground-level to the top of the upper hopper, and the section is oblong. The metal part is 2 ft. 6 in. by 1 ft. 2 in. at the top, and the brick part is 3 ft. 8 in. by 1 ft. 10 in. at the bottom; the metal part is 12 to 15 ft. in length, and the brick part is 19 ft. long. In its newest form, the Henderson retort has two toothed rollers, 3 ft. 6 in. in length, at the bottom to support the shale column and to maintain a downward movement of the shale, thereby regulating the speed of the discharge of spent shale into the hopper below. The original form of this type was provided with a single delivery roller and was smaller; it put through 35 cwt. of shale per 24 hr., while the 1901 retort is capable of retorting 4½ tons per 24 hr.

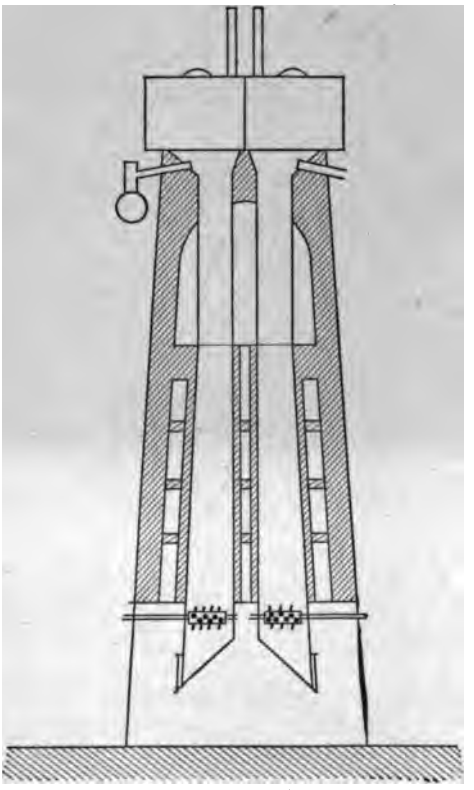


FIG. 313.—The Henderson retort.

Each retort is provided with a malleable-iron hopper on the top, having a capacity of 54 cu. ft. and capable of holding 18 hr. supply of shale. The products are conducted off in a 2-ft. branch pipe at the bottom of the top hopper.

The retort is heated by gas made from dross coal in gas producers, which method is found to be advantageous in ma-

[1] English Patent 6726, 1889.

[2] English Patent 26647, 1901.

nipulating and regulating the heats, besides saving fuel. The increased length and capacity of this type over the Young and Beilby retort resulted in giving the shale longer exposure to the distillation temperature, diminished wear and tear, and increased the yield of ammonia.

Four of these retorts are arranged in an oven, and sixteen ovens constitute a bench. At the Broxburn crude oil works, three and a half benches are in operation (Figs. 314 and 315).



FIG. 314.—Two benches of Henderson retorts at Broxburn, Scotland.

The Young and Fyfe retort[1] is used by the Young's Company; it is merely a remodelled Young and Beilby type, as the improvements were applied to the old retorts in use. Each retort is composed of four sections; namely, a hopper redistillation chamber at the top, a metallic section, a firebrick chamber, and a combustion chamber of large capacity at the bottom. The combustion chamber is not externally heated, like the other sections, but receives the spent shale from the firebrick chamber in

[1] English Patents 13665, **1897**; and 15238, **1899**.

a red-hot condition. The top hoppers are provided with rocking shafts, to which are attached rods or chains, to ensure the regular passage of the shale; and the continuous discharge at the bottom is maintained by a kind of roller.

The Young and Fyfe retort is by no means comparable in efficiency with either the Henderson or Bryson type.

The Crichton or Philipstoun retort was used by James Ross and Company, at Philipstoun. It was built on the principle of the Young and Beilby retort, and consisted of 18 ft. 3 in. of brickwork and an iron part 10 ft. in length. As in the case of many of the shale retorts that have been designed, the Crichton type



FIG. 315.—The crude oil works of the Broxburn Oil Company, Ltd., showing the Henderson retorts, condensers, and spent shale dump.

differed from the others mainly in the mechanical appliance at the base of the retort for removing the spent shale. This appliance consisted of two transverse shafts which worked into a plain boss at the back of the hopper, and passed through an easy-working stuffing-box at the front to the outside of the bench of retorts. The outer ends of the shafts were carried on an iron bearer with cod and coyer, and on the end of each shaft there was an arm connected with links to a hand lever. Each shaft was fitted with a set of grippers, and the discharge of spent shale was regulated by their manipulation. The retorts were drawn every 6 hr., and the throughput was about the same as in the old Henderson retort. The shale in the retort was under perfect

control, but such a type was much more expensive to operate and did not pay on a large scale.

*Retort Condensers.*—The oil and water vapors leave the retorts by an outlet pipe, usually about 8 in. in diameter, and enter a common main, generally about 30 in. in diameter. This main conveys the gases into a water heater, a tower in which water for the steam boilers is heated in pipes, and then into large upright air-cooled condensers, formed of light cast-iron spigot and faucet pipes, 4 in. in diameter, fitted with cast-iron chests and resting on them (Fig. 316). Two hundred ft. of condenser pipe are required for every ton of shale put through per 24 hr. The crude oil and ammonia water collected from the condensers

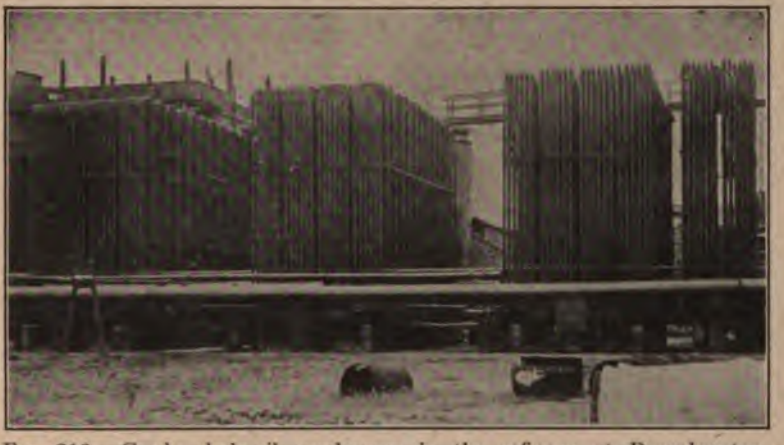


FIG. 316.—Crude shale-oil condensers in the refinery at Pumpherston, Scotland.

are run into a separator, where they are separated, while the uncondensible gases pass into exhausters, which continually maintain a slight pressure on the retorts. The gases then pass into a vertical water-scrubber, where the last traces of ammonia are removed; and next into a naphtha scrubber, where the gas is washed with mineral oil to absorb the naphtha. These scrubbers are generally 5 ft. in diameter and 30 ft. in height.

The efficiency of tower scrubbers for removing ammonia and light hydrocarbons from the uncondensible retort gases depends on three factors: the height of the towers; the extent to which the vapor is split up in its passage through the vessels; and the regular distribution of the absorbing material, which is now

generally wooden chequer work, although coke was used for many years. The theoretical scrubbing limit is never attained in the ammonia scrubbers, and the practical limit is reached when the gas contains not more than 0.5 grain of ammonia per 100 cu. ft.; about 3 lb. of ammonium sulphate per ton of shale put through is recovered. In the naphtha scrubbers it is possible to reduce the illuminating power of the uncondensible gas to about one-half a candle. An intermediate oil (sp. gr., 0.84 to 0.856) is used for scrubbing, and about 2 gal. of light spirit (0.73) are recovered from the scrubbing oil per ton of shale retorted. The number and sizes of these towers are dependent on the number of retorts in operation; but for three benches of



FIG. 317.—Spent shale "bing," Broxburn, Scotland.

retorts (192), three naphtha scrubbers and two ammonia scrubbers are necessary. The scrubbing oil is denuded of naphtha by steaming it in a vessel containing a series of plates and cups with corrugated edges. The denuded mineral oil is used continuously after cooling.

After the retort gas is freed from all condensible and absorbable matters, it is caught by a fan, which forces it under a few inches pressure into the main which supplies the burners at the bottom of the retort flues.

It will be seen that the results of the distillation of shale in retorts are as follows: (1) Spent shale, which is removed from the bottom hoppers and conveyed by hutches to the dumps (Fig. 317); this residue is of no value, although it has been used

in brick-making, road-making, and has been suggested as a material for the manufacture of alum. Spent shale contains about 2.5 per cent. of fixed carbon, and is essentially an aluminum silicate; 80 to 85 per cent. of the raw shale put into the retorts is sent to the waste heaps as spent shale. (2) Permanent gas, which is used as fuel in the retorts. (3) Crude oil, which is pumped into tanks and then refined. (4) Naphtha, which is recovered as above described. (5) Ammonia water, which is distilled for the manufacture of ammonium sulphate.

*Ammonium Sulphate Plant.*—It was some time after the distillation of shale had been started in Scotland before the value

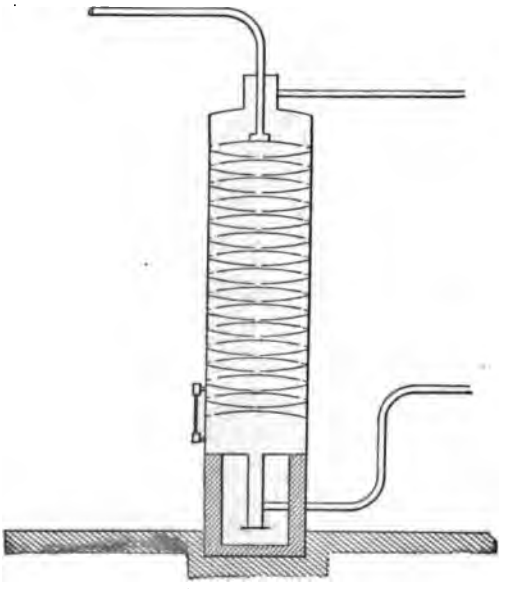


FIG. 318.—The Beilby ammonia column-still.

of the water from the retorts was discovered. Robert Bell, of Broxburn, ascertained its commercial possibilities in 1865 and placed ammonium sulphate on the market in May of that year.

*Stills.*—The method which was first used for expelling ammonia from the ammonia water was to heat the latter in horizontal boilers and conduct the gas into vessels containing sulphuric acid, called cracker boxes. It was found, however, that considerable loss of ammonia occurred from the inability

to drive off all the ammonia contained in the water, and accordingly, in 1882, tower stills were introduced.

The Beilby ammonia column-still was patterned after the Coffey alcohol still, and was the first tower-still used. In this still, the ammonia water entered at the top and was deprived of most of the ammonia, before it overflowed through an outlet at the base of the still, by the action of 20-lb. pressure steam, which passed upward and carried the ammonia vapors with it. The descending water and the ascending steam were forced to travel over a zigzag course, and were thereby brought into intimate contact, by means of concave and convex plates containing openings. The action of this still was continuous (Fig. 318).

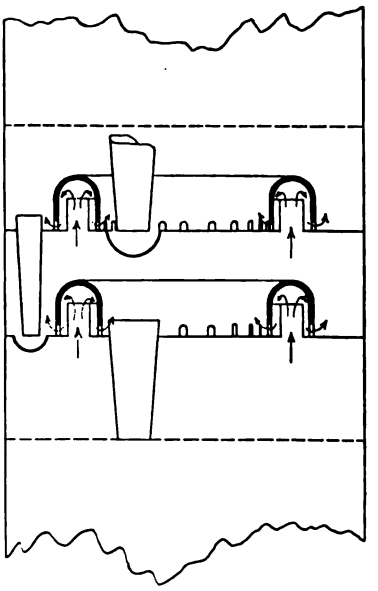


FIG. 319.—Two trays of the Henderson ammonia column-still.

Nearly all the ammonia stills now in operation are based on the Henderson type.[1] In this column-still the ammonia water is run in at the top, and flows from tray to tray, where it is acted on by 30-lb. pressure steam, blown in at the bottom of the stills. The ammonia water is consequently boiled, and the volatile ammonia compounds are expelled in a gaseous state. The stills of this type are generally 22 ft. in height and 5 ft. in diameter, and contain eleven trays (see Fig. 319). The capacity of such a still is 45,000 gal. of ammonia liquor per day.

The gaseous ammonia compounds are conducted into sulphuric acid: first into sulphuric acid recovered from the refinery tars, the ammonium sulphate thus produced being obtained by boiling down the solution; and second, the rest of the gas is passed into a cracker box of fresh chamber sulphuric acid. Wilton's form of cracker box is now widely used. The sulphuric acid flows into the cracker box in a constant stream in lead pipes, with

[1] English Patent 15836, 1885.



FIG. 320.—The sulphuric acid plant of the Broxburn Oil Company, Ltd., at Broxburn, Scotland.



FIG. 321.—The ammonium sulphate plant of the Broxburn Oil Company, Ltd., at Broxburn, Scotland.

sulphuric acid and caustic soda, but is subjected to destructive distillation. This distillation is carried out in pot stills of 2,000 gal. capacity, made of cast iron, which stills are 3 ft. 6 in. to 4 ft. 6 in. deep, and 8 ft. 6 in. in diameter at the top; the top, which is made of steel, is generally 8 ft. 8 in. in diameter by 6 ft. in depth. Each still has a condensing worm 225 ft. in length, made of 4-in. cast-iron pipe, immersed in water in a tank. Before the distillate passes from the condenser to the receiver, it is conducted into a separator box, which is provided with a water outlet at the bottom and an oil outlet higher up. In



FIG. 323.—Crude oil stills in the refinery at Pumpherston, Scotland.

this box the water resulting from the condensation of the steam used in the distillation is separated from the oil distillate. The stills are first heated externally for about 10 hr. to expel all the water contained in the oil, then steam is gradually introduced. The steam used is more or less superheated, and it serves to prevent decomposition, to carry the oil vapors over, and to lower the boiling point of the oil. The quantity of steam used is variable, but when the heavier oils are coming off, the distillate is usually accompanied by 20 per cent. of condensed water. The crude oil is run down to dryness in about a day, and the steam is

not shut off until about 3 hr. after the close of the distillation. The still is then allowed to cool for a day, and the still coke is removed; this material represents about 3 per cent. of the crude oil. The pot stills are run about three times per week. The naphtha, which is collected during the first stage of this distillation, has a specific gravity of about 0.74; and the remainder of the distillate is run into one tank and is known as "once-run oil." There is also some permanent gas produced in this distillation; it is used for fuel. The Broxburn Company has arrangements at its refinery whereby the uncondensed gases from the condensers connected with the crude oil boilers and crude oil residue stills, are collected and used. The gases from the residue



FIG. 324.—Condensers in the refinery at Pumpherston, Scotland.

stills are mostly due to the decomposition of a part of the oil; these gases do not condense to a liquid form under working conditions, but are of value either as illuminants or as fuel. The Broxburn Company obtains about 60,000 cu. ft. of good illuminating gas per day, or about 1¼ cu. ft. for every gallon of crude oil put through. This gas is used for lighting the works and village nearby, which were formerly supplied with gas from coal carbonizers. This company also obtains light naphtha by passing the gas through a water lute; it is made into motor spirit. This naphtha comes over with the overflow water of the hydraulic main, and is separated in an ordinary separator and collected in a receiving tank.

In refining crude shale oil, noxious gases are evolved. From

the condensers connected with the crude oil stills, hydrogen sulphide escapes, and during the subsequent refining and fractionations gases containing hydrogen sulphide are also formed, especially in the residue stills. The Young's Company uses iron oxide purifiers for absorbing the hydrogen sulphide.

In the distillation of 50,000 gal. of crude oil for the production of "once-run oil," twenty-four pot stills and five boiler stills are required. The latter are made of steel, and are for continuous working only; they are generally 30 ft. long and 8 ft. 6 in. in diameter. Boiler stills of this size require a condensing area of 850 sq. ft. In the Henderson system of continuous distillation,[1] the boiler stills are 19 ft. in length and 7 ft. in diameter. The methods of continuous refining now in use in Scotland are based on this system.[2]

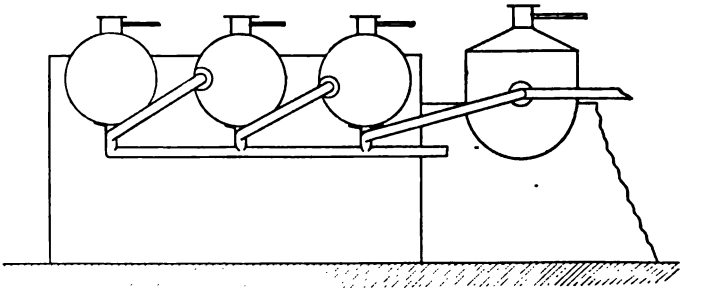


FIG. 325.—Connected boiler stills of the type in use at Broxburn, Scotland.

The "once-run oil" from the crude oil distillation is first settled free from water, and is then pumped into an acid agitator, where it is treated with sulphuric acid. The agitator is first charged with the oil and agitation is started, then the acid is added and the agitation is continued for a period of from 15 min. to over an hour, depending on the grade of the oil. The mixture is then allowed to settle for about a day, and the tar is drawn off at the bottom. The oil is then pumped from the acid agitator into the soda agitator, where it receives the caustic soda treatment. It is allowed to settle for about 8 hr. after the second treatment with caustic soda, and is then pumped to a storage tank to supply the first stage oil stills. The agitation

[1] English Patents 540, 1883; 13014, 1885.

[2] HENDERSON's process of continuous distillation has also been employed in the fractionation of the petroleum from Upper Burma, at Rangoon.

is performed by means of air in an agitator from 6 to 9 ft. in diameter and 10 to 15 ft. in depth; the air is blown in under a pressure of from 6 to 10 lb. per square inch, and serves to mix the oil and acid and oil and caustic soda thoroughly in its passage to the surface.

The treated "once-run oil" is then distilled in the first stage oil stills; it is called "green oil," and is fractionated into naphtha, light oil, heavy oil, and heavy oil and wax. The residuum is coked in pot stills. In a refinery having a working capacity of 50,000 gal. of crude oil, seventeen pot stills and six boiler stills are used at this stage. The light oil and heavy oil are pumped

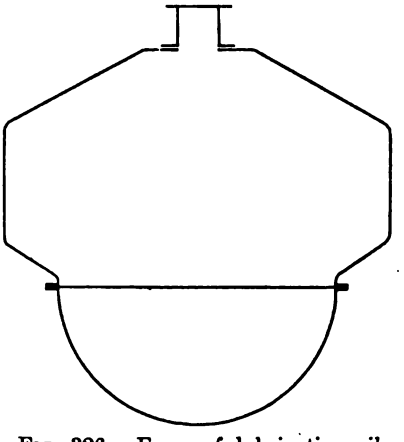


FIG. 326.—Form of lubricating oil still used in the Scottish shale-oil refineries.

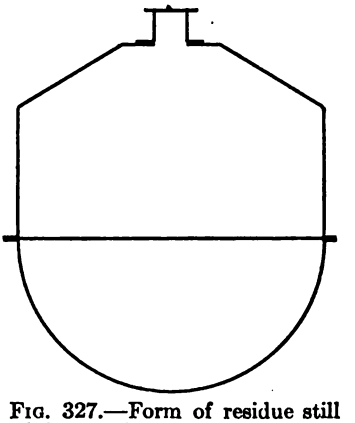


FIG. 327.—Form of residue still used in the Scottish shale-oil refineries.

into separate agitators, and are treated with sulphuric acid and caustic soda, for the removal of the acid and basic tars.

The heavy oil resulting from the above distillation is distilled in boiler stills, fractionating into burning oil, gas oil, and heavy oil. The latter is added to the heavy oil and paraffin from the first-stage distillation, while the gas oil is cooled and pressed in the paraffin house to separate into soft paraffin and 0.850 gas oil. The burning oil is treated with sulphuric acid and caustic soda, and is distilled along with the light oil distillate of the first stage, fractionating into naphtha, burning oil (0.785), burning oil (0.800), burning oil (0.810), and gas oil. The latter is combined with the gas oil from the heavy oil distillation. The heavy oil and paraffin are cooled and pressed in the paraffin

house (see below), producing "blue oil" and hard paraffin scale. The former is refined with sulphuric acid and caustic soda, and is then distilled over caustic soda in boiler stills, fractionating into lubricating oils. The first lubricating oil distillate is cooled and pressed, giving 0.865 oil and soft wax; and the second distillate is given the same treatment, which results in 0.885 oil and soft wax. The lubricating oil is then refined in pot stills holding 2,200 gal., and the soft paraffin wax is added to the sweatings from the hard paraffin scale produced from the heavy oil and paraffin. The sweatings are resweated, form-



FIG. 328.—Stock tanks at the refinery of the Pumpherston Oil Company, Ltd.

ing 100°F. m.p. wax and 110°F. m.p. wax, and the wax from the resweating of the hard scale gives 115°F. m.p. wax and hard wax. The naphtha from the towers connected with the condensers of the retorts is mixed with the naphtha from the first-stage stills, and is treated with acid and caustic soda in agitators provided with means for mechanical agitation. It is then distilled into 0.730 and 0.740 naphtha, and a residue of light oil, which is generally refined with the "green oil."

*Separation of the Paraffin.*—The separation of the paraffin wax of various melting points has been referred to, but a more detailed account of the methods employed is of interest.

The heavy oil is first cooled in tanks set in open sheds and is then further cooled with freezing machines. The latter are generally ammonia absorption machines, in which a solution of calcium chloride is cooled and placed in a tank containing alternate compartments—wide ones for the oil and paraffin, and narrow ones for the brine solution. The oil and paraffin lie in these compartments for many hours, and solid paraffin crystallizes out. Beilby designed a cooler in which the paraffin lay undisturbed until sufficiently cooled, but such a method required much time for crystallization and necessitated a large plant. The Henderson form of cooler[1] is so arranged that a larger throughput may be effected. It is provided with a scraper, which removes the chilled mass of paraffin from the cold plate and permits the warmer material to move to the cold sides of the tank. Some works use dry ammonia gas, liquefied by pressure, and utilize the cold by passing the paraffin mixture through pipes which are chilled by the evaporating ammonia gas. This produces more sudden chilling, and causes some paraffin to thicken in an amorphous state.

From the cooling machines, the paraffin is broken up by machinery and is pumped through filter presses. The paraffin is collected in the press, while the oil flows into its own tank. When the cooling is carried out by means of ammonia gas, without the interposition of the brine, many more filter presses are necessary. The paraffin wax from the filter presses is further squeezed in cloths in hydraulic plate presses, and is called paraffin scale. The heavy oil separated from the solid wax is known as "blue oil;" the refining of this oil has been referred to above.

The crude paraffin was formerly refined by repeated crystallization from naphtha, but this is now accomplished by sweating. In the naphtha treatment the oily matters were removed by dissolving the paraffin in naphtha and allowing it to crystallize out. This operation was generally conducted three times, and the loss of naphtha amounted to about 200 gal. per ton of paraffin refined. The paraffin was freed from the adherent naphtha by being melted in a closed iron vessel, where it was exposed to a current of steam at 20 lb. pressure for 48 to 60 hr. The paraffin was finally decolorized by agitation for 1½ hr. with animal charcoal, in pans, with a horizontal mixer.

[1] English Patent 9557, 1884.

In the sweating process, which is now generally used, naphtha is not employed, the paraffin being simply exposed to such a temperature that the softer paraffin is melted and runs away with the oil. In carrying out the process, the crude scale was formerly melted, and heated to about 180°F., in order that water and other impurities might be separated, after which it was run into pans of 1 to 2 gal. capacity and then allowed to solidify. The cakes were afterward placed on cocoa fiber mats on inclined shelves and ovens, which were heated by steam pipes to about 30°F. below the melting point which the finished product was required to have. The portion which ran out was again treated in the same way and sweated at a lower temperature, and the drainings from it were cooled and pressed to obtain oil and scale, the latter being either worked up with the crude scale mixed with a portion of the intermediate wax or worked alone.

In 1886, Tervet and Alison patented a cooling and sweating arrangement for treating large quantities of paraffin. It consisted of a cooling and sweating chamber, each formed in three sections so that three qualities of wax might be treated.[1] In 1887, Tervet patented another sweating apparatus, in which it was claimed that first-class wax could be obtained from crude scale in one operation.[2]

In the sweating process patented by Norman M. Henderson,[3] which has been widely employed, a chamber about 52 ft. by 13 ft. by 10 ft., having steam pipes for heating it, and enlarged doors and ventilators which may be opened for cooling it, is fitted with a number of superimposed horizontal trays, about 21 ft. by 6 ft. 6 in. wide, resting on transverse heating pipes. Each of these trays is supplied with a horizontal strainer of wire gauze of about 16 meshes to the inch. The bottoms of the trays communicate by short pipes with nozzles constructed to work with worm wheels on a vertical shaft. Before charging the trays, the diaphragm, or strainer, is covered with about ½ in. of water, which prevents the melted wax from running through the diaphragm. The crude solid paraffin from the filter presses, after being melted in the tank, is pumped through the vertical charging pipes, and through the small cocks on to the surface of the water, to fill the trays; and when it is solidified, the water is run off, the

[1] English Patent 8756, **1886.**
[2] English Patent 4, **1887.**
[3] English Patents 1291, **1887**; and 11799, **1891.**

lake of paraffin resting on the gauze, and the doors and venticators are closed. The stove is then heated for a considerable time to 80°F., and finally to the temperature at which the sweating is to take place, and the liquefied impurities are drained off until the overflowing paraffin sets on a thermometer bulb at 130°F. The various liquids separated in the refining of the wax are drawn off through the nozzles into hoppers on the standard pipes, which lead them into horizontal pipes, through which they are passed to suitable receivers. When the sweating is completed, the remaining wax is melted by increasing the temperature and is run off through the same outlets into a tank, from which it is pumped for treatment with bone-black, as in the older processes. The stoves used in this process are built of brick, and have double doors of iron and wood, generally with sawdust between. Each stove is fitted with two sets of nine pans each, and will take from two to three charges of 14 tons each per week, according to circumstances. Larger capacity stoves are also in use. At the Pumpherston refinery there are twenty sweating houses for refining the paraffin wax; each house contains two sets of nine pans each.

Henderson also designed an improved apparatus for sweating paraffin wax, which is said to be superior to his older process. In the new process the paraffin is sweated in cells; a sweating house for an installation of 144 cells being 50 ft. long by 14 ft. wide by 16 ft. high. The full charge of paraffin wax in the cells is 32 tons, and the cells may be charged and run twice a week. Four such installations of 144 cells will give a throughput of 276 tons per week. At the Broxburn refinery, a number of these installations are in operation.

The cost of a refining plant is about £11,000 per 1,000,000 gal. of crude oil run per year, and a refinery to deal with 10,000,000 gal. of oil per year will cost approximately £200,000, everything included. The cost of labor and everything in and for the refinery is three farthings to one penny per gallon of crude oil put through. Three classes of workmen are employed in the refineries: stillheadmen (three for each shift), two assistants to the stillheadmen, and six firemen for each shift.

**Products of Scottish Shale-Oil.**—1. The *permanent gases* produced by retorting are used for fuel. The composition of these gases varies considerably, but the retort gas from Bryson retorts, using Pumpherston shale, generally contains 50 to

60 per cent. of hydrogen and about 20 per cent. of carbon dioxide.

2. *Shale naphtha* usually consists of 60 to 70 per cent. of olefines and other hydrocarbons acted on by fuming nitric acid, the other constituents being principally of the paraffin series. It is completely volatile at ordinary temperatures, and is obtained in different specific gravities and boiling points, according to requirements; for instance, 0.660 for gasoline and 0.690 for motor spirit. Ordinary naphthas, the specific gravities of which range from 0.72 to 0.75, are used for lighting purposes, and as solvents and cleansing agents.

3. *The burning* or *lamp oils* are transparent and nearly colorless. They are used for lamps of many designs, and the specific gravities vary from 0.78 to 0.83. The following are the flash-points of the principal Scottish burning oils:

| | | |
|---|---|---|
| Young's "Crystal" | 128°F. | |
| Pumpherston "Pearline" | 126°F. | All of 18 c.p. |
| Broxburn "Petroline" | 126°F. | |
| Oakbank "Kerosene" | 126°F. | |

Scottish oils have been free from lamp accidents since their manufacture. They are serviceable for continuous burning lamps, for buoys and lightships, and may be used for combustion in oil engines.

4. *Intermediate Oils.*—The specific gravity of these oils varies from 0.84 to 0.87, and the flash-point is generally greater than 150°F. They are used for gas making, and at 800° to 900°C. a good Scottish intermediate oil will yield 1,200 c.p. per gallon; it will be seen, therefore, that the gas produced is of high illuminating power, so they are largely used for enriching coal and water gas as well as for combustion in oil engines.

5. *Lubricating Oils.*—These oils are made with a specific gravity varying from 0.865 to 0.910, and are employed for lubricating purposes, either alone or mixed with vegetable or animal oils. It is claimed that shale lubricating oils do not decrease so rapidly in viscosity by heating as many other mineral lubricating oils.

6. *Paraffin* is used in vast quantities for candle-making, also for water-proofing, metal protection, insulating purposes, and a variety of well-known uses. The usual grades of Scottish paraffin have the following melting points: 130°, 125° to 127°, 118° to 120°, and 110° to 112°F.

7. *Still grease* is the amorphous distillate from the end of the crude oil and heavy oil distillations. It is employed for grease making.

8. *Still coke* is the residue left in the still on running crude oil to dryness. This coke represents, in the case of the best crude oil, about 3 to 4 per cent. of the oil; but in the case of the less pure oil, or where the distillation has not been properly conducted, it may amount to 8 to 10 per cent. of the crude oil. It is used in residences as a fuel, as a smokeless fuel for yachts, as carbon for electrical purposes, and for making moulders' blacking.

9. *Ammonium sulphate* is used as a fertilizer by agriculturists; it is especially useful for growing sugar beet. Ammonium sulphate solutions have also found application in the preparation of fire extinguishers.

10. *Liquid Fuel.*—The acid and basic tars obtained in the refinery are used as liquid fuel for the stills, together with the dregs and residues unfit for other purposes. It is probable that these tars would be valuable as wood preservatives, fluxes for mineral smelting, and for coating iron pipes to prevent the formation of rust. The crude shale-oil produced in Scotland, which has a calorific value of 18,217 B.t.u., has not been employed for fuel purposes, although some experiments were conducted at Woolwich Dockyard in England by the British Admiralty as early as 1866, with the view of testing the value of shale-oil as a substitute for coal in raising steam in marine boilers.[1]

**Profits on a Ton of Shale.**—In 1910, the average cost of mining and manufacturing products from 1 ton of shale in Scotland was given as 8s. 3d.; and the net profit on the products of a ton of shale was stated to be 3s. 4d. In 1882, the profit on a ton of shale amounted to 3s. 7d., while in 1897 the profit was 2s.

The regulation of wages, railway rates, and market prices of the various products, is controlled by an organization composed of representatives of the various Scottish shale-oil companies In 1887, it was proposed to change into an oil syndicate or trust, but this was never effected. Only two strikes have occurred in the history of the industry: one in 1887, from July to October, and another in October, 1903.

[1] On fuel oil from shale, see SELWYN-BROWN, *Eng. Mag.*, **50** (1916), 913.

## SHALE-OIL INDUSTRIES OF OTHER COUNTRIES

The oil-shales in the Wolgan and Capertee valleys, New South Wales, Australia, have been developed by the Commonwealth Oil Corporation, of London. About 20,000,000 tons of oil-shale have been proved in the properties of this corporation, and there are presumably 30,000,000 tons more. The mines and works are located at Newnes, Hartley Vale, Torbane and Ultimo Road, all in New South Wales; and about sixty products are manufactured from "kerosene shale," which yileds up to three and one-half times as much oil as the Scottish shales.[1]

In 1900, a plant was erected at Orepuki, New Zealand, for the distillation of New Zealand shale and the manufacture of products

[1] In a pamphlet accompanying the New South Wales exhibit in the Franco-British Exhibition, London, 1908, the shale being from the Wolgan and Capertee valleys, the yield of gas from this shale was stated to be 17.560 cu. ft. per ton, with an illuminating power of 48.52 standard candles. The yield of crude oil was said to be 101 gal., of sp. gr. 0.877. The results by fractionation were given as follows:

| | Per cent. |
|---|---|
| Naphtha (motor spirit) | 8.48 |
| Illuminating oil | 19.50 |
| Gas oil | 15.87 |
| Lubricating oil | 22.80 |
| Scale | 6.42 |
| Residue | 3.28 |
| Tar, etc | 24.65 |
| | 101.00 |

On the oil-shale industry of Queensland, see BALL, *Queen. Gov. Min. J.*, Dec. 15, **1915.**

[6] A. B. GRIFFITHS (*Chem. News*, **49**, 107) reported that a sample of paraffin shale from near the Golabara River in the western part of Serbia yielded on distillation 2 per cent. of a semi-solid hydrocarbon, similar in appearance to ozokerite.

[7] See DUNLOP, *Rept. Dept. Mines N. Z.*, **1900**, C3, 52.

[8] The oil-shales of New South Wales, Australia, have been described in the following contributions: CLARKE, *Quart. J. Geol. Soc.*, **22**, 439; GREENWELL and BINNEY, *Trans. Manch. Geol. Soc.*, **11**, 63; ANDRAE, *Verh. nat. Ver. Preuss. Rheinl.*, **32**, Sitz., 5; DAWKINS, *Report Brit. Assn.*, **1886**, 643; SKEY, *Annual Report Col. Mus. Lab. N. Z.*, **23**, 50; **25**, 56; **29**, 19; **31**, 10; and PETRIE, *J. Soc. Chem. Ind.*, **24**, 996.

[9] The richest Joadja Creek shales contain but 0.28 per cent. nitrogen.

[10] See WOODRUFF and DAY, *loc. cit.*; and DEBEQUE, *Eng. Min. J.*, **99**, 773.

TABLE LVI.—YIELDS OF SCOTTISH AND OTHER SHALES

| Shale | Yield per long ton | |
|---|---|---|
| | Crude oil in gallons | Ammonium sulphate in pounds |
| Scottish shales | | |
| Airdrie shale | 33 | 6 |
| Westfield shale | 25 | 10 |
| Monkland shale | 32 | 6 |
| Oakbank wee shale | 36 | ..... |
| Oakbank big shale | 22 | ..... |
| M'Lean shale | 38 | ..... |
| Lower wild shale | 19 | ..... |
| Oakbank new shale | 21 | ..... |
| Barracks shale | 19 | ..... |
| Dunnet shale | 7–46 | 14–34 |
| Pumpherston No. 1[1] | 18 | 55 |
| Pumpherston No. 2 | 17 | 60 |
| Pumpherston No. 3 | 20 | 52–67 |
| Pumpherston No. 4 | 20 | 60 |
| Pumpherston No. 5 | 18 | 60 |
| Raeburn shale | 55 | 14 |
| Mungle shale | 35 | 30 |
| Fells shale | 26–40 | 20–35 |
| Broxburn gray shale | 20–33 | 34–41 |
| Broxburn curly shale | 19–33 | 11–38 |
| Broxburn seam shale | 10–50 | 7–40 |
| Other shales | | |
| New Brunswick, Canada, shale | 30–51 | 67–111 |
| Kimmeridge shale, Dorsetshire, Eng.[2] | 50–67 | 38 (maximum) |
| Buxière-les-Mines shale, France[3] | 70–80[4] | 5–7[5] |
| Alexinatz shale, Serbia[6] | 43–46 | ..... |
| Orepuki shale, New Zealand[7] | 20–40 | ..... |
| Hartley seam shale, N. S. W.[8] | 14, 60, 150 | Low[9] |
| Northwestern Colorado shale[10] | 0.3–100 | 0.4–40 |

[1] In the works retorts, the Pumpherston shales yield on the average 20 gal. of crude oil and 50 lb. of ammonium sulphate per ton.

[2] In this connection, see WILLIAMS, *J. Chem. Soc.*, **7**, 97; *Phil. Mag.*, (4), **8**, 209; also, BURTON GREEN'S "Kimmeridge Shale; Its Origin, History and Uses," London, **1886.**

[3] For information concerning the French shale-oil industry, see MAGNIER'S "Nouveau Manuel," Paris, **1867**; CHESNEAU, *Ann. Mines*, (9), **3**, 617; MIRON, "Les Huiles Minérales," Paris, **1897**; and BERTRAND, *Compt. rend.*, **126**, 1677.

[4] Liters per 1,000 kg.

[5] Kilograms per 1,000 kg.

therefrom. The low-grade shale and high development charges resulted in this company discontinuing its work. Oil-shales also occur in Gaspe, New Brunswick, and Nova Scotia, Canada; in the Ronda district in southern Spain; in Tasmania; in Austria-Hungary; in the Machada plain, Turkey; in South Africa, and in Gasa Land, Mozambique, and Orange River Colony, Africa;[1] on the coast of Brazil;[2] in Natal;[3] and in Italy.[4] The brown-coal tar industry of Saxony, Germany, resembles the shale-oil industry in the methods used in the distillation of the lignite, but is in reality a separate industry.[5] Pyropissite, which affords 62 per cent. of paraffin on dry distillation, was at one time distilled in Saxony.

[1] On oil-shale prospects in South Africa, see CRAIG, *Board of Trade J.*, Mar. 19, **1914.**

[2] Concerning the oil shales of Brazil, see CAMERON'S "The Bituminous Deposits of the Camamu Basin, Province of Bahia, in the Brazilian Empire," London, **1884**; and BRANNER, *Trans. Am. Inst. Min. Eng.*, **30** (1901), 537. A plant capable of retorting 50 tons of shale per day was erected ten years ago in Brazil, but was unsuccessful.

[3] DUNSTAN, *Bull. Imp. Inst.*, **1903**, i, 74.

[4] MURRIE, *J. Soc. Chem. Ind.*, **4**, 182; and PULLE, *Ind. chim. min. e met.*, **2** (1915), 209.

[5] See SCHÄDLER'S "Technologie der Fette und Oele," Leipzig, **1883**; SCHULTZ'S "Die Chemie des Steinkohlentheers," Braunschweig, **1890**; LUNGE'S "Die Industrie des Steinkohlentheers und Ammoniaks," Braunschweig, **1900**; GRÄFE'S "Laboratoriumsbuch für die Braunkohlenteer-Industrie," **1908**; and SCHEITHAUER-SALTER'S "Shale Oils and Tars and their Products," **1913.**

## CHAPTER XVIII

## A GLOSSARY OF BITUMENOLOGY

The glossary presented in this chapter contains a list of the important bituminous minerals and products, including oxygenated hydrocarbons; the terms used in naphthology, and the available trade-names of the products of petroleum. Bibliographic data have been included in connection with the definition of many of the subjects.

In collating the terms and definitions the authors have drawn from all available sources of information and from their own experience. Generally speaking, no attempt has been made to uniformize terms which are variously defined, for such standardization is rather the work of a body of experts.

**Acetaldehyde in Petroleum Products.**—On the occurrence of acetaldehyde in petroleum products, see ROBINSON, *J. Soc. Chem. Ind.*, **18** (1899), 232.

**Acetone from Lubricating Oils.**—On the preparation of acetone from lubricating oils, see GUISELIN, *Compt. rend. Cong. intern. Pétrole, sess.* 3, **2** (1910), 561.

**Acid Tar.**—See pages 460, 579 and 841.

**Adeps Petrolei.**—See *Petrolatum*.

**"Adepsine Oil."**—A name for liquid petrolatum, *q.v.*

**"Ader Wax."**—Crude ozokerite (*q.v.*) in leafy masses.

**Adipic Acid from Naphtha.**—On the formation of adipic acid from the naphtha fraction of Russian petroleum ether which boils at 80°C., see ASCHAN, *Ber.*, **32** (1899), 1769; and cf. page 805.

**Adipocerite.**—A synonym for *hatchettite*, *q.v.*

**Aerogene Gas.**—The gas produced by the system of carbureting air devised by VAN VRIESLAND. This system is installed at Breukelen, Holland, for both street- and house-lighting.

**Aeroplane Oil.**—One grade is a white, straight-reduced viscous neutral oil having a gravity of 32¾° to 34°Bé., a flash-point of 415°F., a fire test of 480°F., a cold test of 20°F., and a viscosity of 185 to 200, Saybolt.

**Aggregate.**—The mineral material, such as sand, gravel, shells, slag, or broken stone, or combinations thereof, with which cement or bituminous material is mixed to form a mortar or concrete. "Fine aggregate" may be considered as the mineral inert material which will pass a ¼-in. screen, and "coarse aggregate" the material which will not pass a ¼-in. screen.

**Air Compressor Oil.**—See *Compressor Oil.*

**Ajkite.**—A resin related to succinite, from Ajka, Hungary (*Bull. Soc. Min.*, **1** (1878), 126). See *Succinite.*

**Albertite.**—A jet-black, pitch-like, brittle hydrocarbon possessing a conchoidal fracture and a specific gravity of about 1.1. It differs from ordinary asphalt in being only partly (about 30 per cent.) soluble in turpentine and in being only imperfectly fused when heated.

On the history of albertite, see MILNER, *J. Min. Soc. Nova Scotia*, **17** (1912), 62.

**"Albolene."**—A proprietary brand of *Petrolatum (White).* "Liquid Albolene" is identical with the product described as *Petrolatum (Liquid).*

**Alexjejevite.**—A resin from the Kaluga Government, Russia (ALEXIEEV, *Verhandl. Min. Ges. St. Pet.*, **29** (1893), 201; KARNOJITSKY, *Z. Kryst.*, **24** (1895), 504).

**Algal Wax.**—Algal wax and its relationship to petroleum has been discussed by WATERS in *Am. Chem. J.*, **28** (1902), 78.

**Aliphatic Hydrocarbons from Polymethylenes.**—According to French Patent 462073, Sept. 2, **1913**, of the STEAUA ROMANA PETROLEUM-GES. M. B. H., polymethylenes or mixtures containing polymethylenes, particularly petroleum residuum or mineral oils of high boiling point, are subjected to destructive distillation, and the products are converted into saturated hydrocarbons by treatment with hydrogen in the presence of a suitable catalytic agent.

**Alkaloid Bases in Petroleum and Paraffin.**—On the occurrence of alkaloid bases in Galician petroleum, see BANDROWSKI, *Monatsh. Chem.*, **8** (1887), 224; and on the occurrence of these bases in paraffin oil, see WELLER, *Ber.*, **20** (1887), 2097.

**Allingite.**—A fossil resin from Switzerland; it is related to succinite (AWENG, *Arch. Pharm.*, **232** (1894); *Jahrb. Min.*, **2** (1896), 254). See *Succinite.*

**"Amalie."**—A proprietary name for liquid petrolatum, *q.v.*

**Amber.**—See *Succinite.*

**Ambrite.**—An amorphous, greasy, yellowish gray oxygenated hydrocarbon which occurs in masses in Auckland, New Zealand; it is insoluble in ether, benzene and chloroform (HOCHSTETTER and v. HAUER, *Verhandl. Geol. Reichs.*, **1861**, 4).

**Ambrosine.**—A yellowish to clove-brown resin found in the phosphatic beds near Charleston, S. C.; it may be a modern resin which has been subjected to the action of salt water (SHEPARD, *Rural Carolinian*, **1**, 311).

**American Paraffin Oil.**—An English term for kerosene of American origin.

**Ammonia Oil.**—An oil suitable for the lubrication of the cylinders of ammonia compressors. Low cold test is essential for this purpose.

**Amyl Hydride.**—This fraction in the distillation of petroleum was introduced as an anesthetic by J. BIGELOW and B. W. RICHARDSON in 1867 (see *Sci. Am. Suppl.*, No. **515**, 8227).

**Aniline-Toluidine Oil from Naphtha.**—OGLOBLIN has discussed aniline-toluidine oil from Caucasian naphtha in *J. Russ. Phys.-Chem. Soc.*, **36** (1904), 680.

**Anthracide.**—On anthracide, see BORICKY, *Sitz. K. böhm. Ges. Wiss.*, **1873**, 2; and EICHLEITER, *Verh. K.-K. Geol. Reichs.*, **1899**, 348.

**Anthraconite.**—The name given to varieties of calcite which are darkly colored by the presence of carbonaceous matter.

On the nature of anthraconite, see SKEY, *Trans. New Zealand Inst.*, **25** (1895), 379.

**Anthracoxenite.**—This is obtained as a black powder from a resinoid material from the coal beds of Brandeisl, near Schlan in Bohemia. The resin is treated with ether: *schlanite* dissolves, while the insoluble portion constitutes anthracoxenite (REUSS, *Ber. Akad. Wien*, **21** (1856), 271; *J. prakt. Chem.*, **69**, 428).

**Anthraxolite.**—Anthraxolite, or anthracitic carbon, has been described by COLEMAN and ELLIS in *Rept. Bur. Mines Ont.*, **1896**, 6, 159. It is a black combustible coal-like substance of varying composition, occurring in Ontario and Quebec.

**Anytin.**—See *Anytoles.*

**Anytoles.**—These are proprietary preparations in which substances like phenol, cresol, volatile oils, camphors, etc., are dissolved in water by means of *anytin*, a substance formed by the action of sulphuric acid on various mineral oils, resin oils, and hydrocarbons. The ammonia salt of the hydrocarbons, containing 10 per cent. of chemically combined sulphur, has been found the most useful solvent. The following are some of the anytoles which have been prepared: *phenolanytole, cresol-anytole, meta-cresol-anytole, creosote-anytole, guaiacol-anytole, benzene-anytole, eucalyptol-anytole, peppermint-anytole, wintergreen-anytole, turpentine-anytole, camphor-anytole, and iodine-anytole*—all containing a good proportion of the active ingredient. It has been claimed for these anytoles that they are superior in bactericidal power to the disinfecting substances they contain. WILHELM KOELZER has strongly commended meta-cresol-anytole in the treatment of erysipelas.

**Aragotite.**—A volatile hydrocarbon, said to be related to *idrialite;* it occurs at the New Almaden and Redington mines, Cal., in bright yellow scales (DURAND, *Proc. Acad. Cal.*, **4** (1872), 218).

This rare California mineral is also described by HANKS in *J. Roy. Micr. Soc.*, **1905**, 673.

**Aromatic Hydrocarbons.**—On the utilization of aromatic hydrocarbons derived from cracked petroleum, see RITTMAN, *J. Ind. Eng. Chem.*, **7** (1915), 1014 and 1019.

**Artificial Asphalts.**—See *Asphalt.*

**Artificial Vaseline.**—Another name for *German vaseline, q.v.*

**Asphalt or Asphaltum.**—Solid or semi-solid native bitumen, solid or semi-solid bitumen obtained by refining petroleums, or solid or semi-solid bitumen which is a combination of the bitumens mentioned with petroleums or derivatives thereof, which melts on the application of heat, and which consists of a mixture of hydrocarbons and their derivatives of complex structure, largely cyclic and bridge compounds.

Asphalt has also been defined as a dark-colored, and more or less viscous to solid hydrocarbon complex, including: (*a*) the easily fusible bitumens often associated with a mineral matrix, not having a "waxy" luster or unctuous feel; (*b*) fusible residuums obtained from the distillation, oxidation, sulphurization, etc., of bitumens.

(*a*) *Natural*—resulting from a slow natural process of metamorphosis, known as natural asphalts ("mineral pitches").

1. Occurring in a fairly pure state.
2. Associated with a mineral matrix (*e.g.*, calcareous, silicious or earthy).

(*b*) *Artificial*—resulting from the distillation, oxidation, etc., of mineral oils; known as petroleum asphalt ("petroleum pitches").

1. Straight petroleum asphalt.
2. Cut back.
3. Blown (oxidized) petroleum asphalt.
4. Dubbs' (sulphurized) petroleum asphalt.

On the nature and origin of asphalt, see RICHARDSON, *J. Soc. Chem. Ind.*, **17** (1898), 13. On the differentiation of natural and artificial asphalts, see MARCUSSON, *Mitt. K. Materialprüf.*, **32** (1914), 419.

See *Albertite, Elaterite, Gilsonite, Grahamite, Impsonite, Nigrite,* and *Wurtzilite.*

**Asphalt-Base Petroleum.**—Asphalt-base oils contain asphalt and no paraffin. They are distilled to asphalt and the distillates are cut according to gravity; such oils do not yield steam-refined cylinder stock or paraffin wax.

See *Paraffin-Asphalt Petroleum.*

**Asphalt Block Pavement.**—One having a wearing course of previously prepared blocks of asphaltic concrete.

**Asphalt Cement.**—A fluxed or unfluxed asphaltic material, especially prepared as to quality and consistency, suitable for direct use in the manufacture of asphaltic pavements, and having a penetration of between 5 and 250. See *Flux.*

**Asphalt-Rock.**—The name applied to a stratum of sandstone or limestone when more or less saturated with asphalt.

**Asphalt Substitutes.**—See artificial asphalts, referred to under *Asphalt.*

**Asphaltenes.**—The components of the bitumen in petroleum, petroleum products, malthas, asphalt cements, and solid native bitumens,

which are soluble in carbon disulphide, but insoluble in naphtha (petroleum spirit). See *Petrolene.*

Concerning asphaltene, see PECKHAM, *J. Frank. Inst.*, **151** (1901), 50.

**Asphaltic.**—Similar to, or essentially composed of, asphalt.

**Asphaltic Fluxes.**—The asphaltic fluxes are differentiated from paraffin fluxes and semi-asphaltic fluxes by a still greater density, nearly that of water, by the absence of hard paraffin scale, and by the fact that the unsaturated hydrocarbons predominate. They yield a larger amount of ash-free residual coke on ignition than other fluxes. Those from California petroleum contain less sulphur than that produced from Trinidad oil.

See *Paraffin Fluxes, Semi-Asphaltic Fluxes* and *Mexican Fluxes.*

**Asphaltite.**—A dark colored, solid, difficultly fusible, naturally occurring hydrocarbon complex, insoluble in water, but more or less completely soluble in carbon disulphide, benzol, etc. On asphaltite in northeastern Nevada, see ANDERSON, *Bull. U. S. Geol. Survey*, No. **380**, 283.

**Asphaltum.**—See *Asphalt.*

**Astatki.**—See *Ostatki.*

**Astraline.**—A Russian petroleum product possessing the specific gravity 0.850–0.860, a flash-point not less than 50°C. (122°F.), and a pale yellowish color.

**"Atoleine."**—A name for liquid petrolatum, *q.v.*

**"Atolin."**—A name for liquid petrolatum, *q.v.*

**Automobile Oils.**—The following viscous neutral oils are in use in the cylinders of gasoline cars:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 30.5°Bé. | 415°F. | 480°F. | 20°F. | 215 |
| 31.0° | 415° | 480° | 20° | 210 |
| 32.0° | 400° | 460° | 20° | 145 |
| 33.0° | 415° | 480° | 20° | 200 |

On automobile oils, see CARLES, *Bull. soc. chim.*, (4), **7** (1910), 589; and WATERS, *J. Ind. Eng. Chem.*, **8** (1916), 587.

A prominent producer has supplied the following information:

"Light" automobile cylinder oil is a light bodied oil, possessing a gravity of 30°Bé., a fire test of 475°F., and a Saybolt viscosity of 210. It is pale in color and runs freely at 15°F. It is recommended for use in cylinders of water-cooled gasoline cars.

"Medium" automobile cylinder oil possesses a gravity of 29.5°Bé., a fire test of 480°F., and a Saybolt viscosity of 265. It is pale in color and runs freely at 15°F. It is recommended for use in the cylinders of air- or water-cooled gasoline automobiles.

"Heavy" cylinder oil is a very thick and viscous oil possessing a gravity of 29.2°Bé., a fire test of 485°F., and a Saybolt viscosity of 310.

It is pale in color and runs freely at 15°F. It is recommended for use in gasoline cars where the cylinders have become worn and run very hot; its use is also recommended in motor cycles and motor boats.

**Automobile Transmission Gear Oil.**—A cylinder stock having a gravity of 25°Bé. and a viscosity of 245 is recommended for automobile transmissions.

**Axle Oil.**—Black lubricating oils, especially *summer black oil*, are said to be satisfactory for axle lubrication.

**Baikerinite.**—A thick tar-like fluid at 15°C. which constitutes 32.61 per cent. of *baikerite*.

**Baikerite.**—A wax-like mixture from seams in a rock in the vicinity of Lake Baikal; it is apparently about 60 per cent. ozokerite.

Baikerite has been described by HERMANN, *Bull. Soc. Imp. Nat. Mosc.*, **30** (1857), 470; and by SCHRÖDER, *Verh. k. russ. min. Ges.*, (2), **36** (1898), 36.

**Bakuin.**—A Russian machine oil, prepared from Baku petroleum; it has been recommended for lubricating heavy machinery because of its high viscosity and great power of resisting cold (*J. Soc. Chem. Ind.*, **3**, 181).

**Bakuol.**—A name given by MENDELÉEFF (*Z. Technik*, **1886**, No. 109; *Chem.-Ztg.*, **1888**, 231) to an illuminating oil, prepared from the oils of Baku by mixing ordinary kerosene of the specific gravity 0.82 to 0.83 and flash-point 20° to 30°C., with the so-called intermediate oil, which possesses a specific gravity of 0.86 to 0.88 and is not inflammable at 100° C. The mixture has a specific gravity of 0.84 to 0.86 and flashes at 50° to 70°C.

**Bakurin.**—A lubricating oil, prepared by mixing 100 parts of crude Baku petroleum with 25 parts of castor oil and 60 to 70 parts of 66°Bé. sulphuric acid. After standing, the mixture is agitated with water, which is then run off, and the oil is treated with either sodium or potassium hydroxide (MÜLLER, German Patent 35141; *Dingler's polyt. J.*, **260**, 240).

**"Bakurol."**—A name for liquid petrolatum, *q.v.*

**Barbados Bitumen.**—Concerning this bitumen, see DUBOIS, *Z. prakt. Geol.*, **7** (1899), 397; EMTAGE, *J. Soc. Arts*, **52** (1904), 367; and REHWAGEN, *Berg-Hütt. Ztg.*, **60** (1901), 467. See *Asphalt*.

**"Barbados Tar."**—The dark green or black petroleum of Barbados, which was formerly widely used in medicine (JAMES' "Medicinal Dictionary," **1745**).

**Barnstaple Oil-Shale.**—The occurrence of mineral oil in Upper Devonian shale at Barnstaple, England, has been described by HALL in *Trans. Devon. Assn.*, **6** (1874), part ii, 547.

**Barracks Shale.**—One of the principal oil-shale seams of Scotland. See page 813.

**Base.**—The artificial foundation of a pavement.

**Bases in Petroleum.**—On the pyridine bases in petroleum, see ZALOZIECKI, *Monatsh. Chem.*, **13** (1892), 498. See also page 7.

**Batching Oil.**—A non-viscous neutral oil of, preferably, 34°Bé. and having a viscosity of 80, Saybolt.

**Bathvillite.**—An amorphous, fawn-brown, opaque, very friable oxygenated hydrocarbon from Torbane Hill, Scotland; it is insoluble in benzol and is related to Torbanehill mineral (*q.v.*). It is described by WILLIAMS in *Chem. News*, **7** (1863), 133.

**Benzine.**—That fraction of b.p. 70°–120°C. obtained in the refining of petroleum. Heptane ($C_7H_{16}$) is the principal constituent of the benzine from Pennsylvania oil. Totally distinct from the aromatic hydrocarbon *benzene*, $C_6H_6$.

Deodorized benzine, the light solvent of the "U. S. Pharmacopœia," has a specific gravity of 0.638–0.660 at 25° (82°–89°Bé.) and a b.p. of 45–60°C. The benzine of some American refiners—that used in the oilcloth and varnish industries—has a specific gravity of 0.729 (62°Bé.); of others, 0.741–0.745. See pages 459, 465, 467, 471, 481, 489, and 503 for full information regarding the production and grades of benzine. Cf. *gasoline* and *naphtha*.

**Benzine, Heavy.**—See *Ligroin*.

**"Benzine-Naphtha."**—A naphtha having a gravity of 70°Bé. It is made by treating and redistilling crude naphtha of the gravity 80° to 68°Bé.

**Benzinum.**—Benzinum is thus described in the "United States Pharmacopœia:" "A distillate from American petroleum consisting of hydrocarbons chiefly of the marsh-gas series."

This useful product of petroleum was first introduced into the "United States Pharmacopœia," 1880; it is obtained in the process of purifying petroleum by fractional distillation, and it is defined in the "Pharmacopœia" as a transparent, colorless, diffusive liquid, of a strong, characteristic odor, slightly resembling that of petroleum, but much less disagreeable, and having a neutral reaction. Petroleum benzine is highly inflammable, and its vapor, when mixed with air and ignited, explodes violently. Specific gravity, 0.638 to 0.660 at 25°C. (77°F.). Insoluble in water; soluble in about 6 parts of alcohol, and readily soluble in ether, chloroform, benzene, volatile oils, and fixed oils, with the exception of castor oil. Boiling point, 45° to 60°C. (113° to 140°F.).

**Benzinum Purificatum.**—Purified petroleum benzine was introduced into the 8th revision of the "United States Pharmacopœia;" it is used mainly as a solvent. The official method of purification is based upon a process devised by BERINGER (*Am. J. Pharm.*, **1890**), which is intended to remove sulphur compounds and other impurities; this method is as follows: 60 c.c. of sulphuric acid are added to 550 c.c. of water; and when the mixture has become cold, it is poured into a bottle having the capacity of about 2 liters. Eight grams of potassium permanganate are then

added and the mixture is agitated until the salt is dissolved; 1 liter of petroleum benzine is then added in four portions, shaking the liquid after each addition. After allowing the liquids to remain in contact for 24 hr., shaking the bottle at frequent intervals, the petroleum benzine is decanted into another bottle of the same capacity. It is then mixed and agitated frequently during several hours with a solution of 2 grams of potassium permanganate in 240 c.c. of water, in which 2 grams of sodium hydroxide have previously been dissolved; following this treatment, the benzine is decanted, the washing with water is repeated, and the purified petroleum benzine is again decanted.

**Benzoline.**—The more volatile portion obtained on redistilling benzine; b.p., about 70°–95°C. Often used as synonymous with *benzine*.

**Berengelite.**—A dark brown, resinous, asphalt-like mineral, soluble in cold alcohol but nearly insoluble in potassium hydroxide, found near Arica, Peru (JOHNSTON, *Phil. Mag.*, (2), **13** (1838), 329).

**Bermudez Asphalt.**—Refined Bermudez asphalt has a specific gravity of 1.071 at 60°F.; it contains 97.22 per cent. of bitumen soluble in carbon disulphide, 1.50 per cent. of ash, 1.28 per cent. of non-bituminous organic matter; and it possesses a softening point of 113°F. and a flowing point of 150°F.

On this asphalt, see *Rept. Operat. Eng. Dept. D. C.*, **1894**, 143; and RICHARDSON, *Pop. Sci. Mon.*, **81** (1912), 19.

**Beta-Jaulingite.**—A brownish yellow resin, obtained from the residue of *jaulingite*, after treatment with carbon disulphide, by the action of ethyl ether.

**Bielzite.**—A brittle, resinous, brownish black mineral from Transylvania; it has a specific gravity of 1.249, and dissolves in considerable part in carbon disulphide and chloroform. It has been described by BENKÖ and JAHN in *Z. Krystal.*, **13** (1887), 68; *Orv.-term. Tud. Ert.*, **1886**, 159.

**Binder.**—(1) A foreign or fine material introduced into the mineral portion of the wearing surface of a pavement for the purpose of assisting the road metal to retain its integrity under stress, as well as, perhaps, to aid in its first construction. (2) The course, in a sheet-asphalt pavement, frequently used between the concrete foundation and the sheet-asphalt mixture of graded sand and asphalt cement.

**Binding Coals.**—Coals which cake on burning.

**Birmite.**—See *Burmite*.

**Bitter-Almond Oil, Artificial.**—WAGNER has described the preparation of artificial bitter-almond oil from petroleum in *Dingler's polyt. J.*, **136** (1855), 311.

**Bitumen.**—A mixture of native or pyrogenous hydrocarbons and their non-metallic derivatives, which may be gases, liquids, viscous liquids, or solids, and which are soluble in carbon disulphide.

*Bitumen* is also defined as a naturally occurring hydrocarbon com-

plex, often associated with a mineral matrix, insoluble in water, but largely soluble in carbon disulphide, benzol, etc. Its color and hardness are variable. On bitumen, see *Min. Ind.*, **25**, 69.

**Bitumen from Bovey.**—See *Retinasphaltum.*

**Bituminous Bound.**—Bonded with the aid of bituminous material. See *Bond.*

**Bituminous Cement.**—A bituminous material suitable for use as a binder, having cementing qualities which are dependent mainly on its bituminous character.

**Bituminous Concrete Pavement.**—A pavement, composed of stone, gravel, sand, shell or slag, or combinations thereof, and bituminous materials, incorporated together by mixing methods.

**Bituminous Macadam Pavement.**—A pavement, having a wearing course of macadam, with the interstices filled by penetration methods with a bituminous binder.

**Bituminous Materials.**—Materials containing bitumen as an essential constituent. In the broad sense, the term "bituminous materials" applies to materials containing mixtures of native or pyrogenous hydrocarbons and their non-metallic derivatives, which may be gases, liquids, viscous liquids, or solids, and which are soluble in carbon disulphide. This definition is still a matter of controversy, but has the sanction of technical use. It is usually limited to the plastic materials derived from natural asphalts, from oxidized petroleums, and from the tars and pitches obtained from the destructive distillation of coal, mineral oil, etc. These materials may be broadly classified as natural asphalts, products of destructive distillation, and oxidized petroleum products. The natural asphalts range from the light fluid malthas consisting of an asphaltic base and a light volatile hydrocarbon, to the hard solid asphalts of the gilsonite type. See ABRAHAM'S "Asphalts and Allied Substances," **1917.**

The tars and pitches may be obtained as the by-products in the manufacture of coal gas, water gas, oil gas, coke, or in any process where carbonaceous matter is burned with a limited supply of air.

The oxidized petroleums are obtained by blowing air through usually heavy petroleum residues at elevated temperatures. The changes produced are complex; but, in brief, part of the hydrogen present in the oil combines with the oxygen contained in the air and passes off as water vapor or steam, so that the per cent. of carbon is increased. The physical character of the oil is changed from a viscous liquid to a more or less rubber-like solid.

All of these materials vary to such an extent with their origin, treatment, method of refining, and final blending that a classification based either on origin, method of production or subsequent treatment is impracticable. This is due in part to the fact that very few asphalts are used in their natural condition; they are treated in a variety of ways and blended with other materials to modify their original character. The

harder asphalts are fluxed with petroleum oils so as to make them more plastic, while the more fluid are frequently heated so as to drive off the more volatile products. The artificial asphalts, obtained by oxidation of petroleum residues, may be modified in the same manner, while the tars and pitches vary with their source, method of production, and subsequent treatment.

**Bituminous Pavement.**—One composed of stone, gravel, sand, shell or slag, or combinations thereof, and bituminous materials, incorporated together.

**Bituminous Sand.**—On the vast resources of bituminous ("tar") sand in northern Alberta, Canada, see *Rept.* **231**, *Mines Branch, Canada Dept. of Mines.*

**Bituminous Shales.**—Argillaceous shales much impregnated with bitumen; or shales containing kerogen (*q.v.*). See *Shale Oil.*

**Bituminous Substance.**—A term applied to (*a*) substances containing bitumens or pyro-bitumens; (*b*) substances resembling either the viscous or solid bitumens, or pyro-bitumens in their solubility and physical properties, *i.e.*, having an amorphous structure and dark color in mass. See *Bituminous Materials.*

**Bituminous Surface.**—In paving, a superficial coat of bituminous material, with or without the addition of stone or slag chips, gravel, sand, or material of similar character.

**Bitusol.**—Trinidad asphalt is said to be a true bitusol—that is to say, dispersed solid colloids in solution in bitumen. See PIERCE, *Met. Chem. Eng.*, **13** (1915), 697.

**"Black Oils."**—Black oils may be either residues from petroleum or from its distillates. They vary widely in character and are used as cheap lubricants, on car wheels and on other places where the higher refined oils are not necessary. Two "black oils" from Pennsylvania petroleum had these properties: gravity, 23° and 26° Bé.; flash point, 450° and 365°F.; fire test, 530° and 425°F.; Saybolt viscosity, 84 at 212°F. and 50 at 212°F.

**"Blaes."**—In Scotland, the brownish oil-shales are interstratified with non-bituminous or less bituminous beds, sometimes of a bluish gray color; these are termed "blaes" by the shale miners. See *"Kingle."*

**"Blandine."**—A name for liquid petrolatum, *q.v.*

**Blanket.**—See *"Carpet."*

**Blau or Blue Gas.**—A mixture of volatile hydrocarbons, such as propane, butane, the pentanes, etc., containing hydrogen and methane, etc., in solution under pressure. The pressure solution is filled into steel cylinders, and is thus available for transport. Blau gas is used for illuminating, heating and power purposes. See HALLOCK, *J. Soc. Chem. Ind.*, **1908**, 550.

**"Bleeding."**—The exudation of bituminous material on the roadway surface after construction.

**"Blown Petroleums."**—Semi-solid or solid products obtained primarily by the action of air upon originally fluid native bitumens, which are heated during the blowing process. See *"Condensed" Oils.*

**"Blue Oil."**—In the Scottish shale-oil industry, the oil produced from heavy oil and paraffin by cooling and pressing for separation of hard paraffin scale; it is refined and fractioned into lubricating oils. See page 836.

**"Bog-Butter."**—See *Butyrellite.*

**Boghead Cannel.**—See *Torbanehill Mineral.*

**Boghead Coal.**—A highly bituminous Scottish mineral containing 60–70 per cent. of volatile matter, from which Young obtained 120–130 gal. of crude oil per ton. It was worked out in 1862.

On the manufacture of coal gas from boghead coal, see FYFE, *J. Frank. Inst.*, **24** (1852), 347 and 399; on the composition of this coal, see FYFE, *Trans. Roy. Scot. Soc. Arts*, **4** (1856), 250; and for a general account of the Torbanehill minerals, see TRAILL, *Trans. Roy. Soc. Edinb.*, **21** (1854), 7.

See *Torbanehill Mineral.*

**Bolt Oil.**—A viscous neutral oil having a gravity of 30°Bé. and a Saybolt viscosity of 220, has been used for nut and bolt threads and is recommended for that purpose.

**Bombiccite.**—A transparent, colorless mineral, found in lignite in Tuscany; it fuses at 75°C., volatilizes at a higher temperature, and is soluble in carbon disulphide, alcohol and ether (BOMBICCI, *Mem. Accad. Bologna*, **9**, 1869; GUARESCHI, *Boll. Com. Geol.*, **2** (1871), 70).

**Bond.**—The combined action of inertia, friction, and of the forces of adhesion and cohesion which aids the separate particles composing a pavement to resist separation under stress. Mechanical bond is the bond produced almost wholly, in a well-built broken-stone macadam road, by the interlocking of angular fragments of stone and the subsequent filling of the remaining interstices with the finer particles.

**"Bottom Settlings" ("B. S.").**—Earthy matter, inert organic matter, or, in the case of paraffin-base petroleum, an emulsion of amorphous paraffin wax and water, which accompanies crude oil. If an emulsion, it may be refined with the petroleum after the removal of water; otherwise it may be eliminated by settling. See p. 447.

**Boulevard Gas Fluid.**—A petroleum distillate which had a gravity of 76°Bé.; it was used for street naphtha lamps.

**Bound.**—Bonded.

**Branchite.**—A mineral from the brown coal of Mt. Vaso in Tuscany; it is colorless, has a specific gravity of 1.044, and is soluble in alcohol. See *Hartite.*

**Brea.**—Sand or soil impregnated with petroleum from seepages, the volatile constituents having evaporated. Brea is associated with petroleum in formations of the Puente Hills district, Cal., and has been used as a road dressing.

**Brick Oil.**—One grade is a pale non-viscous neutral oil, or light-bodied lubricating oil, possessing a gravity of 34°Bé., a flash-point of 340°F., a

fire test of 400°F., a cold test of 20°F., and a viscosity of 80, Saybolt. The product described under *paint oil* is also known as brick oil.

**British Oil.**—A rubefacient liniment composed of oil of turpentine, linseed oil, oil of amber, oil of juniper, and petroleum.

**Brown-Coal.**—See *Lignite.*

**Broxburn Oil-Shale.**—A Scottish shale which averages 5¼ ft. in thickness and is found at Addiewell, Seafield, Oakbank, Broxburn, Hopetoun, and Philipstoun; it yields 23–35 gal. of crude oil and 35–40 lb. of ammonium sulphate per ton. See page 913.

**Brücknerellite**—A substance ("geo-retinic acid") found associated with leucopetrite (BRÜCKNER, *J. prakt. Chem.*, **57** (1852), 1); it was reported to have the composition $C_{34}H_{44}O_2$.

**"B. S."**—See *Bottom Settlings.*

**"B. S. Oil."**—A term applied to crude oil tank residues. On employment of these, see page 632.

**Bucaramangite.**—A resin resembling amber in color, but insoluble in alcohol and yielding no succinic acid (BOUSSINGAULT, *Ann. chim. phys.*, **6** (1842), 507; *Compt. rend.*, **96**, 1452). See *Succinite.*

**Buffing Oil.**—A 31°Bé. viscous neutral oil is said to be satisfactory in buffing.

**Burmite.**—A fossil resin, resembling amber, but harder and tougher; it occurs in Upper Burma (HELM, *Rec. Geol. Survey India*, **25** (1892), 180; **26**, 31, 61). See *Succinite.*

**Burning Oil.**—See *Kerosene.*

**Butane.**—On the butanes in American petroleums, see MABERY and HUDSON, *Proc. Am. Acad.*, **32** (1897), 101.

See *Cymogene.*

**Butyrellite.**—A white or yellow waxy substance found in certain of the Irish and Scotch bogs. It has been described as a mineral, but is of undoubted animal derivation.

On the chemical composition of butyrellite, see MACADAM, *Min. Mag.*, **6** (1885), 175.

**Byerite.**—A caking bituminous coal from Middle Park, Colo. It resembles albertite in the large amount of gas and oil which it yields upon distillation, but is heavier (1.323) and is insoluble in carbon disulphide, etc. (*Am. J. Sci.*, **9** (1875), 146).

**Byerlyte.**—The name given by F. X. BYERLEY to the product obtained by his patented process (United States Patent 524130, Aug. 7, 1894) of manufacturing asphaltic substances from petroleum by the prolonged exposure to high temperature and the action of air. See page 629.

**Caking Coal.**—Caking coals are bituminous coals which soften and become pasty or semi-viscid in the fire. This softening occurs at the temperature of incipient decomposition; on increasing the heat, the volatile products are expelled, and a coherent, cellular, or fritted mass of coke is left. On coking and caking coals, see VIALL, *Power*, **43** (1916), 432.

**Camphenes.**—Camphenes of the general formula $C_nH_{2n-4}$ have been found in the distillate from the asphalt of Pechelbronn (BOUSSINGAULT, *Ann. chim. phys.*, **64**, 141; **73**, 443) and in that from the asphalt of the Val de Travers (VÖLCKEL, *Ann.*, **87** (1853), 143).

**Canadol.**—A light petroleum ether ("light ligroin") of the specific gravity 0.650–0.700, which has been used for the production of local anesthesia by spraying, and as a solvent.

**Cannel Coal.**—A variety of bituminous coal, often caking, compact, little or no luster, and breaking with a conchoidal fracture and smooth surface. It was formerly distilled in Scotland, where its occurrences graduate into coaly oil-shales. On the destructive distillation products of cannel coal, see *Bull. School of Mines, Univ. Mo.*, **3** (1917), No. 4.

**Caoutchouc.**—"Australian caoutchouc" has been described by DYER in *J. Bot.*, (6), **1** (1872), 103, 338. "Derbyshire caoutchouc" is described in *Ann. Mus. Hist. Nat. Paris*, **1** (1799), 261.

On mineral caoutchouc, see MORRIS, *Proc. Acad. Nat. Sci. Phila.*, (3), **7** (1877), 131.

See *Helenite* and *Elaterite*.

**Car Oil.**—Car oils are usually black lubricating oils of the same general character as *summer black oil*. Lubricants for car axles must be cheap, and some railroad specifications require a gravity of 29°Bé., a flash-point of 325°F., and a cold test of 5° to 15°F.

**Carbenes.**—The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements, and solid native bitumens, which are soluble in carbon disulphide, but insoluble in carbon tetrachloride. See *Asphaltenes* and *Petrolene*.

**Carbon Black.**—A name for *lampblack*.

**Carbonite.**—Carbonite, or native coke, occurs at the Edgehill mines, near Richmond, Va.; it is more compact than artificial coke and some varieties afford bitumen. See WURTZ, *Trans. Am. Inst. Min. Eng.*, **3** (1875), 457.

**Carbon Oil.**—A name for kerosene.

**Carbopetrocene.**—PRUNIER (*Bull. soc. chim.*, (2), **31** (1879), 293) isolated from petrocene (*q.v.*) hydrocarbons which he termed carbozene, carbopetrocene, and thallene. See *Carbozene*.

**Carboxylic Acids.**—According to MARKOVNIKOV (*J. Russ. Phys.-Chem. Soc.*, **19** (1887), 156), as much as 5.25 per cent. of oxygen occurs in the fraction of Caucasian petroleum boiling between 220° and 230°C.; he considers the acids to be carboxylic acids, derived from the naphthenes of the petroleum. ASCHAN (*Ber.*, **23** (1890), 867; **24** (1891), 2710) has confirmed this view.

**Carbozene.**—Hydrocarbons termed carbozene, carbopetrocene and thallene, possessing formulas ranging from $(C_4H_2)_n$ to $(C_7H_2)_n$, where $n$ is a variable higher than 4, have been isolated by PRUNIER (*Bull. soc. chim.*, (2), **31** (1879), 293) from the so-called petrocene (*q.v.*).

**Carpet.**—A bituminous surface of appreciable thickness, generally

formed on top of a roadway by the application of one or more coats of bituminous material with gravel, sand or stone chips added.

**Castor Machine Oil.**—An oil much used for the lubrication of axles, journals and harvesters, usually supplied in three grades. It is the solution of an aluminum soap in paraffin or light neutral oil.

**Cedarite.**—A fossil resin resembling amber, somewhat widely distributed in the alluvium of the Saskatchewan River in Canada (*Jahrb. Min.*, **2** (1898), 212). See *Succinite*.

**Cement.**—An adhesive substance used for uniting particles of other materials to each other. Ordinarily applied only to calcined "cement rock," or to artificially prepared, calcined, and ground mixtures of limestone and silicious materials. Sometimes used to designate bituminous binder used in bituminous pavements, when the expression "bituminous cement" (*q.v.*) is understood to be meant.

**Cement Waterproofing Oil.**—See *Concrete Waterproofing Oil*.

**Cerasin.**—See *Ceresin*.

**Ceresin.**—Ceresin is prepared by heating ozokerite (*q.v.*) with sulphuric acid, with constant stirring, and decolorizing with charcoal; the product is then treated with volatile solvents to extract the contained ceresin. Ceresin is used as a substitute for beeswax. It occurs in commerce in the form of thin round masses several inches in diameter, harder than wax, of a dazzling white appearance; inodorous, and transparent at the edges. The melting-point is between 75° and 80°C.

**Chemawinite.**—A resin related to succinite (*q.v.*), occurring on a beach on Cedar Lake, near the mouth of the north Saskatchewan; it has a specific gravity of 1.055, its color varies from pale yellow to dark brown, and it is soluble to the extent of 21 per cent. in absolute alcohol (HARRINGTON, *Am. J. Sci.*, **42** (1891), 332).

**"Cherry Coal."**—The cherry coal, or soft coal, of England, is a non-caking coal which ignites well and burns rapidly.

**Cholesterin in Petroleum.**—On the alleged occurrence of cholesterin in the laevo-rotatory fractions of certain petroleums, see STEINKOPF and WINTERNITZ, *Chem.-Ztg.*, **38** (1914), 613; and pages 30 and 114.

**Christmatite.**—A butyraceous, greenish yellow to wax-yellow hydrocarbon from Wettin, Saxony; it has a specific gravity of below 1 and is soft at 55° to 60°C. See FLECK'S "Steinkohlen Deutschl.," 1865, **1**, 37.

**Chrysene.**—On chrysene and its derivatives, see ADLER, *Ber.*, **12** (1879), 1889; and LAURENT, *Compt. rend.*, **5** (1837), 718.

**Claroline.**—A mineral oil possessing a specific gravity of 0.8667 at 15°C., a viscosity of 4.4° Engler at 20°C., a flash-point of 152°C., and an ignition-point of 270°C. It is a solvent for natural gases.

**Cleansing Oil.**—A naphtha possessing a boiling point of 120° to 150°C. and a specific gravity of 0.722 to 0.737 at 15°C.

**Cloustonite.**—A mineral related to asphalt, occurring in patches in blue limestone and in blue flags at Inganess, Orkney. It is soluble in

benzol and at a red heat gives 47.8 per cent. of "illuminating gas" (HEDDLE, *Min. Mag.*, **3** (1879), 222).

**Coal Mine Drivers' Oil.**—See *Drivers' Oil.*

**Coal Oil.**—This term has been applied to the following: the crude oil obtained by the destructive distillation of bituminous coal; that distillate obtained from such a crude oil which was used for illuminating purposes; crude petroleum; and kerosene.

**Coal Tar.**—The mixture of hydrocarbon distillates, mostly unsaturated ring compounds, produced in the destructive distillation of coal. Raw coal tar is composed of light oils, pyridine bases, phenols, naphthalene, anthracene, heavy oils, etc. It varies greatly in composition, and may be divided into retort gas tar and oven gas tar, according to the method of production. See G. LUNGE'S "Coal-Tar and Ammonia," New York, **1916.**

**Coal-Tar Naphtha.**—The light oil produced in the distillation of coal tar. For an excellent description of coal tar and its distillation products, see DODGE in ROGERS and AUBERT'S "Industrial Chemistry," 2d edition.

**Coal-Tar Oils.**—It is a common practice in this country, in the distillation of coal tar, to fraction as light oil until the distillate commences to sink in water, and as heavy oil or creosote oil from that point to pitch.

Heavy coal-tar oils are the dead anthracene oils, heavier than water and having the odor of creosote; they are completely soluble in ethyl alcohol. On the differentiation of coal-tar oils from mineral oils, see VALENTA, *Chem.-Ztg.*, **30** (1906), 266.

**Coal-Tar Pitch.**—The residuum from the distillation of coal tar. Most of the tar is run to soft pitch with a melting point between 60° and 80°C.

**Coat.**—(1) The total result of one or more single surface applications. (2) To apply a coat. See *"Carpet."*

**Coke.**—Petroleum coke usually shows the following composition: Volatile and combustible matter, 5 to 10 per cent.; fixed carbon, 90 to 95 per cent.; ash, from a trace to 0.3 per cent.; sulphur, from 0.5 per cent. to 1 per cent. On account of the purity of petroleum coke it has found application in metallurgical processes and in making battery carbons and carbon pencils ("electric carbons").

On petroleum coke, see DOELTZ, *Chem.-Ztg.*, **30** (1906), 585; LANGBEIN, *ibid.*, 1115; LIDOV, *J. Russ. Phys.-Chem. Soc.*, **14** (1882), 323; and PRUNIER and VARENNE, *Bull. soc. chim.*, (2), **33** (1880), 567.

**Coke, Native.**—See *Carbonite.*

**Coke-Oven Tar.**—Coal tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

On bituminous-coal coke, see BYROM and CHRISTOPHER'S "Modern Coking Practice," **1910**; and LEWES' "Carbonization of Coal," **1912.**

**Coking Coals.**—See *Caking Coals.*

**"Combustible Shale."**—See *Tasmanite.*

**Compounded Oils.**—Mineral lubricating oils are often mixed with seed oils; for instance, "blown" rapeseed oil or "blown" cottonseed oil are used to increase viscosity. Then, too, varying amounts of an animal oil are frequently added to mineral cylinder oils, and artificial viscosity is sometimes imparted to some of the less viscous mineral lubricating oils by the addition of rosin oil or of aluminum oleate or palmitate. See *Castor Machine Oil.*

**Compressor Oils.**—A straight-reduced viscous neutral oil of the gravity 30.5° or 31°B6. and having a viscosity of 215 or 210, Saybolt, is said to be satisfactory for air compressors. Compressor oils should have a solidifying point of below −20°C. and a flash-point of 140° to 180°C. They are sometimes artificially colored to a violet tint.

The light spindle oil described under *ice-machine oil* has also been recommended for air compressors.

**Concrete Form Oil.**—One grade is a non-viscous neutral oil possessing a gravity of 34°B6., a flash-point of 340°F., a fire test of 400°F., a cold test of 20°F., and a viscosity of 80, Saybolt.

**Concrete Waterproofing Oil.**—*Summer black oil* is said to be satisfactory for waterproofing cement. On the grade of oil required for this purpose, see *Bull. No.* **46**, *Office of Public Roads, U. S. Dept. Agr.*; and *Bull. No.* **230**, *U. S. Dept. Agr.*

**"Condensed" or "Blown" Oils.**—1. The bitumens prepared from the residual fluxes by treatment with sulphur or air at high temperatures, are said to be short or lacking in ductility and to be non-adhesive. The effect of such treatment is to produce a material which may be looked upon as dehydrogenated and condensed as far as its molecular structure is concerned. "Condensed" or "blown" oils all yield much ash-free residual coke, and the effect of the process is to reduce to a marked extent the percentage of saturated hydrocarbons of which they consist and the solubility of the substance in naphtha. Without regard to the character of the petroleums in which they originate, the resulting products resemble one another quite closely.

2. Vegetable oils, such as cottonseed oil, which are thickened by oxidation, for compounding marine cylinder oils, are also known as "blown" oils.

See *Asphalt*, *Hydroline*, *Obispo*, *S. O. Co. Binder*, and *Gulf Binder*, also *"Blown Petroleums."*

**Consistency.**—The degree of solidity or fluidity of bituminous materials.

**Coorongite.**—A south Australian *elaterite*, or mineral caoutchouc (*q.v.*).

On coorongite, see BOODLE, *Bull. Roy. Bot. Gardens, Kew*, **1907**, 145; CUMMING, *Proc. Roy. Soc. Vict.*, **15** (1903), 134; JACKSON, *Pharm. J.*, **31** (1872), 763 and 785; and MORRIS, *Proc. Acad. Phila.*, **1877**, 131.

**Copaline.**—See *Copalite.*

**Copalite.**—An oxygenated hydrocarbon resembling copal, from the

blue clay of Highgate Hill, near London, England (AIKIN's "Manual of Mineralogy," **1815,** 64; JOHNSTON, *Phil. Mag.*, **14** (1839), 87).

**Cordage Oil.**—A non-viscous neutral oil usually identical with *concrete form oil* in properties.

**Core Oil.**—The grade described as *paint oil*, 36°Bé. gravity, compounded with corn, soya bean or linseed oil, is said to be satisfactory as a core oil.

**"Cosmolene," or "Cosmoline."**—Cosmoline, or *Unguentum Petrolei*, is a trade-name for petrolatum. See page 631.

On cosmoline, see LEMBERGER, *Proc. Am. Pharm. Assn.*, **1874,** 384; MABERY, *Proc. Am. Acad.*, **40** (1904), 361; MILLER, *Am. J. Pharm.*, (4), **3** (1872), 534; **4** (1874), 1.

**Cottonseed Oil Adulterant.**—The product described as *paint oil* has been used in the adulteration of cottonseed oil.

**Courtzilite.**—A form of asphaltum allied to gilsonite (*q.v.*). See *17th Ann. Rept. U. S. Geol. Survey*, **1895-6,** part III, 752.

**"Crackene."**—An aromatic hydrocarbon ($C_{24}H_{18}$), obtained by KLAUDY and FINK (*Monatsh.*, **21** (1900), 118) from the "red pitch" condensing in the stills during the cracking of Austro-Hungarian petroleum. It formed yellow crystalline scales with green fluorescence, m.p., 308°C., and was quite similar to benzerythrene.

**Crank-Case Oil.**—Viscous neutral oils of 30.5° and 32°Bé. gravity are recommended for gas engine crank cases. A mixture of 30 per cent. of 26°Bé. cylinder stock and 70 per cent. of this with 32°Bé. viscous neutral oil has been used for steam crank cases; and an oil of gravity 26° to 27°Bé., flash 455°F., and Saybolt viscosity 100 at 212°F., is said to have proved generally satisfactory.

Crank-case oils should emulsify but little with water.

**Cream Separator Oils.**—The following non-viscous neutral oils have been used and recommended for the lubrication of hand separators:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 34.0°Bé. | 340°F. | 400° | 20°F. | 80 |
| 34.5° | 340° | 400° | 20° | 75 |
| 35.0° | 340° | 400° | 20° | 70 |

The following viscous neutral oils have been recommended for lubricating steam separators:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 30.5°Bé. | 415°F. | 480°F. | 20°F. | 215 |
| 31.0° | 415° | 480° | 20° | 210 |
| 32.0° | 400° | 460° | 20° | 145 |

**Cristo-Grahamite.**—Grahamite (*q.v.*) from the Cristo mine, Huasteca, Mexico.

**"Crude."**—A name for crude petroleum.

**Crude Mineral Oil.**—A name for crude petroleum.

**Crude Oil.**—A name for crude petroleum. See page 1.

**Crusher Oil.**—An oil which has been recommended for crushers contains 50 per cent. of *summer black oil* and 50 per cent. of 26°Bé. steam-refined cylinder stock.

**"Crysmalin."**—A proprietary name for liquid petrolatum, *q.v.*

**Cumene.**—De la Rue and Müller have found cumene ($C_9H_{12}$) in Rangoon petroleum (see *Proc. Roy. Soc.*, **8** (1857), 221).

**Curly Shale.**—A Pumpherston oil-shale. Its thickness is about 6 ft., and it yields 20 gal. of crude oil and from 60 to 70 lb. of ammonium sulphate per ton. See page 813.

**Curve Grease.**—A grease adapted for use on railroad curves.

**Cut-Back Products.**—Petroleum or tar residuums which have been fluxed, each with its own or similar distillates.

**Cutting Oils.**—On cutting oils, see Copeland, *Met. Chem. Eng.*, **17** (1917), 25.

**Cycloheptanes, Cyclohexanes, and Cyclopentanes.**—On the presence of cyclic hydrocarbons in the fractions 48°–50°C., 50°–51°C., and 90.5°–98°C., obtained from Caucasian naphtha, see Markovnikov, *Ber.*, **30** (1897), 974 and 1221. On the advantages offered by Caucasian naphtha as a source of cyclic compounds, see Markovnikov, *Ann.*, **301** (1898), 154; **302** (1898), 1.

**Cylinder Stocks.**—Steamed cylinder stocks, oils which are used in the lubrication of steam cylinders, for which purpose they are frequently compounded with fatty oils, are the steam residues from paraffin-base crude oils. They vary in gravity from 20° to 27°Bé., in flash from 650° to 475°F., in viscosity from 350 sec. to 100 sec. Saybolt universal viscosimeter at 210°F., and in cold test from 30° to 60°F. They are dark green in color and contain varying amounts of asphaltic matter, depending upon the quality of crude oil refined and the care exercised in refining. These oils are often refined by filtration through fullers' earth or bone-black, which process lowers the specific gravity and the cold test, but reduces the viscosity; all these changes depend upon the extent of filtration. Filtered cylinder stocks may be recognized by their transparency (cf. *unfiltered stocks*). See pages 469, 474, 476, and 482.

Valve or cylinder oils are often compounded with tallow oil or neats-foot oil.

Certain eastern refiners produce the following filtered cylinder oils:

| Gravity | Flash-point | Fire test | Saybolt viscosity |
|---|---|---|---|
| 29°Bé. | 450°F. | 500°F. | 90 |
| 28° | 500° | 550° | 120 |
| 27° | 540° | 600° | 140 |
| 26¼° | 550° | 615° | 165 |
| 27° | 550° | 610° | 150 |

Five grades of "steam-refined cylinder stocks" are usually produced from Pennsylvania petroleum; the properties of these are as follows:

| | | | | | |
|---|---|---|---|---|---|
| Gravity, degrees Bé..... | 26.0 | 25.5 | 25.0 | 24.5 | 24.5 |
| Saybolt viscosity at 212°F..... | 160.0 | 168.0 | 200.0 | 225.0 | 250.0 |
| Cold test, degrees F..... | 40.0 | 40.0 | 30.0 | 30.0 | 30.0 |
| Fire test, degrees F..... | 600.0 | 610.0 | 635.0 | 650.0 | 675.0 |
| Flash-point, degrees F..... | 535.0 | 545.0 | 570.0 | 590.0 | 600.0 |

On the valuation of cylinder oils, see RICHARDSON and HANSON, *J. Soc. Chem. Ind.*, **24** (1905), 315.

**Cymogene.**—A petroleum product possessing a specific gravity ranging between 0.590 (108°Bé.) and 0.636 (90°Bé.); it has usually a boiling point of 0°C. and is nearly pure butane. It has been used as a local anesthetic, in certain types of refrigerators and in the manufacture of ice. See *Butane* and *Rhigolene.*

**Dam-Shale.**—A Scottish oil-shale which averages from 3 to 6 ft. in thickness. See page 812.

**Danforth's Oil.**—The trade name of a petroleum distillate possessing a specific gravity of 0.69 to 0.70 and boiling at 80° to 110°C. It is used as a solvent and for burning in vapor stoves.

**Dead Oils.**—Oils with a density greater than water, which are distilled from tars.

**Deblooming Agents.**—Mono-nitronaphthalene ($C_{10}H_7NO_2$) and yellow coal-tar dyes are sometimes added to mineral oils to mask the fluorescence. Oils treated with nitronaphthalene become darker on keeping. See *Nitronaphthalene.*

**Decane.**—Normal and secondary decane have been found in Galician petroleum (LACHOWICZ, *Ann.*, **220** (1883), 188).

**"Deeline."**—A liquid petrolatum.

**Dehydrated Tars.**—Tars from which all water has been removed.

**Deodorized Naphtha.**—Two grades of deodorized naphtha are marketed by Pennsylvania refiners: 62°–63°Bé., used by dyers; and 55°–58°Bé., used by paint and varnish manufacturers.

**Diabantite.**—Diabantite is described by HAWES in *Am. J. Sci.*, (3), **9** (1875), 454.

**Die Oil.**—The following neutral oils are employed in the lubrication of steel dies:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 30½°Bé. | 395°F. | 450°F. | 30°F. | 240 |
| 34° | 340° | 400° | 20° | 80 |

**Di-isopropyl in Baku Benzine.**—On the presence of di-isopropyl in benzine, see ASCHAN, *Ber.*, **31** (1898), 1801.

**Dinite.**—An inodorous, tasteless, fragile mineral having the appearance of ice, but with a yellow tinge, and very soluble in ether and carbon disulphide; it was found in a lignite deposit at Lunigiana, Tuscany.

**Distillate Fuel Oil.**—Fuel oil of heavy gravity. One having a gravity of 34°Bé. (7.11 lb. per gallon), has been found to contain 20,250 B.t.u. per pound.

See *Fuel Oil.*

**Docosane.**—Docosane ($C_{22}H_{46}$) has been separated from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565).

**Dodecane.**—Dodecane ($C_{12}H_{26}$) has been isolated from Pennsylvania petroleum. See MABERY, *Proc. Am. Acad.*, **32** (1897), 121.

**Dopplerite.**—An amorphous, elastic or jelly-like, brownish black oxygenated hydrocarbon found in peat beds, near Aussee in Styria, at Bad Gonten, Appenzell, and at Obburgen, Switzerland; it has a specific gravity of 1.089 and is insoluble in alcohol and ether.

Regarding dopplerite, see CLAESSEN, *Chem.-Ztg.*, **22** (1898), 523; COOPER, *Eng. Min. J.*, **32** (1881), 103; DEMEL, *Monatsh.*, **3** (1882), 763; LEWIS, *Proc. Am. Phil. Soc.*, **20** (1882), 112; and KENNGOTT, *Jahrb. k.-k. geol. Reichs.*, **1** (1850), 303.

**Dopplerite of Deicke.**—A grayish, earthy, plastic oxygenated hydrocarbon from a peat bed at Finkenbach, Switzerland (DEICKE, *Berg.-Hüttenm.-Ztg.*, **17**, 383). It is not the dopplerite described above.

**Dorsetshire Bituminous Shales.**—Deposits of bluish gray slaty clay, containing volatile matter, and interstratified with thin beds of highly bituminous shale, occur in Dorsetshire, England. See *Kimmeridge Shale.*

On the destructive distillation of this shale, see WILLIAMS, *J. Chem. Soc.*, **7** (1854), 97; *Phil. Mag.*, (4), **8** (1854), 209.

**Drivers' Oil.**—Either the grade described as *paint oil* or a non-viscous neutral oil of 34°Bé. has been recommended for coal mine drivers' oil.

**Dry Cleaning Gasoline.**—Gasoline of 66° to 68°Bé. or deodorized naphtha (*q.v.*) of 58° to 60°Bé. is used for dry cleaning. See *Naphtha.*

**"Dry" Natural Gas.**—Natural gas which does not contain an appreciable amount of readily condensible gasoline. It is usually not intimately associated with petroleum. See page 440. Cf. *"Wet" Natural Gas.*

**Drying Oils from Petroleum.**—See page 804.

**Dunnet Shale.**—An oil-shale, from 4 to 12 ft. in thickness, found at West Calder, Oakbank, Pumpherston, Duddingston, Newliston, and Burntisland, Scotland; it overlies the Lower Dunnet shale which is worked at Oakbank, and yields from 24 to 33 gal. of crude oil per ton.

**"Durbon."**—A "natural carbon" paint, manufactured from shale. Shale is heated in air-tight retorts, and both the distillate of oil and the black residue are used in the production of "Durbon."

**Dust-Laying Oils.**—Crude oils, heavy asphalt oils, tars, solutions of petroleum asphalt in gas-oils, liquid asphalt, and emulsions of oils and water, have been used for laying dust on roads. The basic idea in the manufacture of dust-laying oils is that the solvent or lighter hydrocarbons will slowly evaporate, leaving the dust particles covered with a sticky adherent film. Petroleum asphalt in solution in oils similar to gas oil is widely used.

See *"Kiton"* and *Westrumite.*

Dust-laying oils used for wood floors and linoleum should be pure mineral oils of high specific gravity, which should not afford a separation of sticky substances in the course of several weeks. See *Floor Oil.*

**Duxite.**—A resin from the lignite of Dux, Bohemia; it fuses at 246°C., has a specific gravity of 1.133, and is near walchowite (Doelter, *Verh. Geol. Reichs.*, **1874**, 145).

**Dyes from Petroleum.**—On the production of aniline dyes from petroleum, see Kharitchkov, *Trudi Bak. Otd. Imp. Russk. Tekhn. Obsch.*, **1905, 1906, 1907;** Mason, *Rept. Cons. U. S.*, **5** (1882), 43; and page 806.

**Dynamo Oils.**—Among the grades of viscous neutral oil used in dynamo lubrication are the following:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 30.5°Bé. | 415°F. | 480°F. | 20°F. | 215 |
| 30.5° | 410° | 465° | 25° | 190 |
| 31.0° | 415° | 480° | 20° | 210 |
| 31.5° | 40 | 450° | 25° | 161 |
| 32.0° | 400° | 460° | 20° | 145 |

Dynamo oil is also termed "dynamo engine oil."

**Dysodile.**—A very inflammable, flexible, slightly elastic, yellow or greenish gray mineral from Melili, Sicily, and from certain German lignite deposits; it has a specific gravity of 1.14 to 1.25.

On dysodile, see Cordier, *J. Mines*, **23** (1808), 275; Delesse, *These anal. Chim.*, **50**, 1843; Church, *Chem. News*, **6** (1862), 122; **34** (1876), 155; Ehrenberg, *Ann. phys. chem.*, **124** (1839), 573; and Stephens, *Proc. Roy. Soc. Tasmania*, **1876**, 5.

**Dysodyl.**—See *Dysodile.*

**Earth Wax.**—See *Ozokerite.*

**Earthy Brown Coal.**—A brown friable mineral, sometimes forming layers in beds of lignite. In general, it is not a true coal, for a considerable part of it is soluble in ether and benzol, and often in alcohol. See *Leucopetrite* and *Bathvillite.*

**"Ebano."**—In 1908, four grades of Ebano Mexican residual pitches were on the market. Ebano "E" contained 99.4 per cent. of bitumen soluble in carbon disulphide, 1 per cent. of paraffin scale, 5.9 per cent. of sulphur, and 19.2 per cent. of ash-free residual coke. Ebano "DX" contained 97.9 per cent. of bitumen soluble in carbon disulphide, 1.3 per cent. of paraffin scale, 6.33 per cent. of sulphur, and 23.9 per cent. of ash-free residual coke. Ebano "D" contained 97.8 per cent. of bitumen soluble in carbon disulphide, 1.3 per cent. of paraffin scale, 6.08 per cent. of sulphur, and 24.9 per cent. of ash-free residual coke. Ebano "B" contained 95.8 per cent. of bitumen soluble in carbon disulphide, 1.9 per cent. of paraffin scale, 6.9 per cent. of sulphur, and 30.5 per cent. of ash-free residual coke.

See *Pitch*.

**Elastic Bitumen.**—See *Elaterite* and *Caoutchouc*.

**Elaterite.**—A variety of bitumen, known as "mineral India rubber," on account of its elastic properties, was found at an early date in the Odin Mine, Derbyshire, and was termed elaterite by Hausmann. It is similar to *coorongite*.

Utah "elaterite" is a massive amorphous dark brown hydrocarbon, which ranges from soft and elastic to hard and brittle. See *Wurtzilite*, of which elaterite is a trade-name.

Elaterite has been described by ALEXIEEV, *Gorn. Jurn.*, **1889**, 1, 361; LISTER, *Phil. Trans.*, **1673**, 6179; MAGUIRE, *Mines and Minerals*, **20** (1900), 398; and MORRISON, *Min. Mag.*, **8** (1889), 133.

**Electrum.**—See *Succinite*.

**Elsass Bitumen.**—On this bitumen, see FAVRE, *Bull. Soc. Sci. Nat. Neuchâtel*, **11** (1878), 122; MIEG, *Bull. Soc. Ind. Mulhouse*, **53** (1883), 83; and SPIELMANN, *Hist. Acad. Roy. Sci., Berlin*, **14**, 105.

**"Emolene White Oil."**—A water-white, odorless oil for medicinal purposes.

**"Emulsicool."**—A cutting compound which, when mixed with water, forms an emulsion. It is said to be especially adapted for cutting and drilling.

**Emulsion.**—A combination of water and oily material made miscible with water through the action of a saponifying or other agent.

**Endecane.**—Endecane ($C_{11}H_{24}$) has been isolated from Pennsylvania petroleum (see MABERY, *Proc. Am. Acad.*, **32** (1897), 121).

**"Engine Distillate."**—"No. 1 Engine Distillate" from California petroleum is water-white and has a gravity of 50°Bé.

"No. 2 Engine Distillate" from the same source possesses a water-white to light amber color and a gravity of 45°Bé.

**Engine Gasoline.**—A gasoline for both stationary and portable engines. See *Motor Gasoline*.

**Engine Oil.**—One grade of viscous neutral oil used in the lubrication of engines has a gravity of 29° to 30°Bé., a flash-point of 400° to 415°F.,

a fire test of 450° to 480°F., a cold test of 20° to 30°F., and a Saybolt viscosity of 180 to 300. See *Machine Oil.*

The grade described as *compressor oil* is used as a high-speed engine oil and in the splash lubrication of engines; and a mixture of 60 per cent. of 32°Bé. viscous neutral oil with 40 per cent. of engine oil has been used in turbines.

While the above specifications are in use, the hydrocarbon oils sold as engine oils may present wide ranges in gravity (23° to 32°Bé.), flash (300° to 400°F.), and viscosity (50 to 400 sec. at 70°F.). Moreover, engine oils are sometimes mixed with animal oils, as lard or whale oil.

**Ether from Petroleum.**—For some time petroleum was used as a source of ethylene for the production of ethyl ether, according to the method of FRITSCHE (see *Chem.-Ztg.*, **33**, 759; United States Patent 475640, Jan. 19, 1897).

**Ethylene.**—Preponderating quantities of the ethylene group have been found in Rangoon petroleum (DE LA RUE and MÜLLER, *Proc. Roy. Soc.*, **8** (1857), 221). According to HÖFER, ethylene has been isolated from North American petroleum.

**Euosmite.**—An amorphous, brownish yellow, brittle oxygenated hydrocarbon from clefts in brown coal at Baiershof, near Thumsenreuth, in the Fichtelgebirge; it has a specific gravity of 1.2 to 1.5, and dissolves easily in alcohol and ether (GÜMBEL, *Jahrb. Min.*, **1864**, 10).

**Eupion.**—On eupion, see REICHENBACH, *J. chem. phys.*, **62** (1831), 129; **66** (1832), 318; *Ann. phys. chem.*, **1832**, 173; **113** (1836), 534; and VÖLKEL, *Verh. Schweiz. Ges. Nat.*, **1850**, 112.

**"Export Oil."**—An illuminating oil for export; it is usually of 44°Bé. gravity, or lighter. That for England is of 73° Abel flash-point and that for Germany has a flash-point of 21° Celsius; both are of standard white quality.

**"Fat."**—Containing an excess. A fat asphalt mixture is one in which the asphalt cement is in excess and the excess is clearly apparent.

**Fatty Acids from Petroleum.**—See page 805.

**Fells Shale.**—This important oil shale, said to be the richest in Scotland, is worked at Tarbrax, Addiewell, Breich, and Seafield; it yields from 26 to 40 gal. of crude oil and from 20 to 35 lb. of ammonium sulphate per ton.

**Fichtelite.**—A white, translucent, brittle, odorless hydrocarbon from peat beds near Redwitz, North Bavaria; it is easily soluble in ether, is soluble in cold nitric acid, and distils without decomposition.

On fichtelite, see BAMBERGER, *Ber.*, **22** (1889), 635; BAMBERGER and STRASSER, *ibid.*, 3361; BROMEIS, *Ann.*, **37** (1841), 304; CLARK, *Ann.*, **103** (1857), 236; **119** (1861), 226; HELL, *Ber.*, **22** (1889), 498; PLZAK and ROSICKY, *Z. kryst. min.*, **44** (1908), 332; SCHMIDT, *Arch. Pharm.*, (3), **6** (1875), 538; and SPIEGEL, *Ber.*, **22** (1889), 3369. Fichtelite has been found in Alabama.

**Fish Oil Adulterant.**—A viscous neutral oil of 32°Bé. gravity and 145 Saybolt viscosity has been employed for fish oil adulteration and substitution.

**Fixed Carbon.**—The organic matter of the residual coke, obtained upon burning hydrocarbon products in a covered vessel in the absence of free oxygen.

**Floor Oil.**—A non-viscous neutral oil having the same properties as *concrete form oil.* Floor compound oil is of the same grade. Such oils should give a good finish and prevent dust from arising.

**"Floridin."**—Fullers' earth from Quincy and Jamieson, Florida, used in decolorizing petroleum products.

On the employment of floridin in the refining of petroleum products, see BENSMANN, *Compt. rend. internat. Cong. Pétrole, sess.* 3, **2** (1910), 695; and on the recovery of paraffin from bleaching earth by extraction, see GREMPE, *Seifenfabrikant,* **35** (1915), 7345.

**Flush Coat.**—See *"Seal Coat."*

**Flux.**—Bitumens, generally liquid, used in combination with harder bitumens for the purpose of softening the latter.

The materials which are termed fluxes are the residuals left after the removal by distillation of the more volatile fractions of various petroleums, until the product is of such a density that it contains little or nothing volatile on heating 50 grams for 5 hr. at a temperature of 325°F. They consist of viscous oils of a character used for fluxing solid asphalts or for direct application to road surfaces as a "carpet coat."

See *Paraffin Fluxes, Asphaltic Fluxes, Semi-Asphaltic Fluxes* and *Mexican Fluxes.*

**Flux Oil.**—A product from asphalt-base petroleum, used by roofing concerns and paving contractors to flux natural asphalts; it is obtainable in various gravities, that of 11° to 14°Bé. being most used.

FLUX OILS, GALLON WEIGHT CHART

| Gravity at 60°F. | Pounds per gallon | Gallons per ton | Gravity at 60°F. | Pounds per gallon | Gallons per ton |
|---|---|---|---|---|---|
| 10 | 8.331 | 240.0 | 18 | 7.885 | 253.6 |
| 11 | 8.273 | 240.0 | 19 | 7.833 | 255.3 |
| 12 | 8.215 | 243.4 | 20 | 7.781 | 257.0 |
| 13 | 8.158 | 245.1 | 21 | 7.730 | 258.7 |
| 14 | 8.102 | 246.8 | 22 | 7.679 | 260.4 |
| 15 | 8.047 | 248.5 | 23 | 7.630 | 262.1 |
| 16 | 7.992 | 250.2 | 24 | 7.581 | 263.8 |
| 17 | 7.939 | 251.9 | 25 | 7.533 | 265.5 |

**Foam Oil.**—*Paint oil,* 45°Bé. illuminating oil, and *mineral seal oil* are used as paper mill foam oils.

**Foliated Coal.**—See *Leaf-Coal.*

**Form Oil.**—See *Concrete Form Oil.*

**"Formolite."**—For the determination of the unsaturated cyclic hydrocarbons in a mineral oil, NASTYUKOV (*Petroleum*, **4**, 1336 and 1397) treats the oil with formaldehyde in the presence of concentrated sulphuric acid, whereby a solid yellow product is separated, for which the name "formolite" has been proposed. See also HERR, *Chem.-Ztg.*, **34** (1910), 893; and RICHARDSON, *J. Ind. Eng. Chem.*, **8** (1916), 319.

**Fossil Copal.**—See *Copalite.*

**Fossil Grease.**—KINAHAN has described fossil grease in the *Athenæum*, **1883**, 780.

**Free-Burning Coals.**—Coals which do not cake on burning.

**Free Carbon.**—In tars, organic matter which is insoluble in carbon disulphide.

**Freezing Machinery Oils.**—See *Compressor Oils* and *Ammonia Oil.*

**Fuel Oil.**—(1) Any product of petroleum used for the production of power or heat. Fuel oils from Mid-Continent petroleum range from at least the distillate series, down to and including any product which can be made liquid by steam heat and for which no better market outlet can be obtained. California fuel oil has a gravity of about 17°Bé.; it has a flash-point of 150°F. and contains about 30 per cent. of asphalt. (2) From another standpoint, fuel oils are the distillates heavier than illuminating oils and lighter than lubricating oils, ranging from 25° to 30°Bé. On the employment of fuel oil, see F. B. DUNN'S "Industrial Uses of Fuel Oil," **1916.**

See *Distillate Fuel Oil* and *Reduced Fuel Oil.*

**Gagates.**—According to DIOSCORIDES and PLINY, gagates occurred at Gagas or Gages, in Lycia (Asia Minor); it was, in part at least, true lignite (*q.v.*). See *Jet.*

**Gas Black.**—A name for *lampblack.*

**Gas Engine Gasoline.**—See *Motor Gasoline.*

**Gas Engine Oil.**—A number of grades of viscous neutral oil are marketed for gas engine lubrication. The following products serve as illustrations:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| Straight reduced.....30.5° | 415°F. | 480°F. | 20°F. | 215 |
| 31.0° | 415° | 480° | 20° | 210 |
| 32.0° | 400° | 460° | 20° | 145 |
| Stock loaded.....30.5° | 395° | 450° | 30° | 240 |

DAVIS ("Friction and Lubrication," 121) states that for large size gas engines an oil having these characteristics is suitable: gravity, 26° to 28°Bé.; flash-point, 400° to 475°F.; viscosity, 250 sec. at 70°F.

**Gas engine oils** should not carbonize at the temperatures attained. See *Automobile Oils.*

**"Gas Fluid."**—See *Boulevard Gas Fluid.*

**Gas-House Coal Tar.**—Coal tar (*q.v.*) produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

**Gas, Natural.**—The gaseous products arising from petroleum wells. See page 438.

**Gas Oil.**—Gas oils are intermediate between burning oil and lubricating oil (b.p. 600°–650°F.). Gas oil from Pennsylvania petroleum usually possesses a gravity of from 34° to 42°Bé., a flash-point of 230°F., a fire test of 270°F., and a cold test of 20°F. As a rule, 1 kg. of gas oil yields about 550 liters of gas, 350 grams of tar, and 50 grams of coke.

For a select bibliography of gas oils and oil gas, see *Technical Paper* 120, *Bureau of Mines*, 12–14.

**"Gasol."**—A product condensed from casing head gas by applying a pressure of 850–900 lb. per square inch at ordinary temperature. It has a specific gravity of 0.5, and 1 lb. of the liquid produces 7 cu. ft. of gas, said to be suitable as a fuel in autogenous welding. See *Machinery*, 21 (1915), 903.

**Gasoline.**—This name has been applied broadly to the lighter products derived from petroleum, ranging in gravity from 50°Bé. to the high gravity products, 90°Bé. and over, which are extracted from the still gases by the compression method.

There are at present on the market types of gasoline produced by several general methods. These may be classified as follows: "straight" refinery or normal gasoline; blended casing-head gasoline; and "cracked" or synthetic gasoline. Normal gasoline exists naturally in petroleum; and "cracked gasoline" is formed by the decomposition of the heavier products. Normal gasoline has a low iodine absorption, whereas gasoline produced by the cracking process may have a high iodine absorption.

The examination of gasoline should include gravity test, and the determination of color, acidity, volatility, and unsaturated hydrocarbons.

Normal gasoline should be water-white in color, of sweet odor, should evaporate without leaving any stain or appreciable odor, and the iodine absorption should be below 5 per cent. Normal gasoline is used, or rather should be, in all dry-cleaning establishments.

"Cracked" gasoline, as the name implies, is likely to fluctuate in properties. Speaking generally, and of its application to use in gas engines, it should show a low initial boiling point, and should have a final boiling point of not higher than 350°F. As long as these conditions are fulfilled, the lowest gravity is the best product.

That fraction of b.p. 40° to 70°C. obtained in the refining of Pennsylvania petroleum, consists largely of pentane and hexane. The grades of gasoline and benzine made from Pennsylvania petroleum vary in gravity from 56°Bé. to 86°Bé.; the eight grades usually marketed are as follows: 60°, 62°, 63°, 64°, 66°, 68°, 70°, and 72°Bé. The gasoline used for automobile purposes varies from 60° to 74°Bé. The heavier

gravities are extensively used for mixing paints and for blending with condensed gas gasoline; the lighter grades are employed in the manufacture of rubber cements and in illumination. Gasoline from California petroleum usually has a gravity of 60°Bé. (See "*Tops.*")

There is some confusion in the use of the names benzine, gasoline and naphtha, but the best practice is to employ the term gasoline for any mixture of light hydrocarbons intended for use in any kind of vaporizer, *i.e.*, to be gasified in a gas machine, gasoline torch, gasoline stove, or automobile carburetor. See *Naphtha.*

**Gear Case Oil.**—A steam-refined cylinder stock often used for gear cases, has a gravity of 25°Bé., a flash-point of 610°F., a fire test of 710°F., a cold test of 30°F., and a Saybolt viscosity of 245.

**Gedanite.**—A resin resembling amber, but not containing succinic acid and less rich in oxygen; it is found on the shores of the Baltic. See *Succinite.*

On gedanite, see HELM, *Arch. Pharm.*, (3), **13,** 503; and ZINCKEN *Berg.-Hütt. Z.*, **39** (1880), 73.

**Geocerain.**—See *Geocerite.*

**Geocerellite.**—A white, brittle, alcohol-soluble oxygenated hydrocarbon which melts at 82°C. (BRÜCKNER, *J. prakt. Chem.*, **57** (1852), 14).

**Geocerite.**—A wax-like, white oxygenated hydrocarbon which melts at about 80°C.; it is soluble in alcohol and is unacted upon by a hot potassium hydroxide solution. It is said to have the composition $C_{28}H_{56}O_2$ (BRÜCKNER, *J. prakt. Chem.*, **57** (1852), 14). Geocerite occurs in the brown coal of Gesterwitz. See *Geomyricite.*

**Geomyricin.**—See *Geomyricite.*

**Geomyricite.**—A wax-like, white mineral, melting at 80°–83°C., and soluble in hot absolute alcohol and ether; its composition ($C_{34}H_{68}O_2$) is near that of certain vegetable waxes (BRÜCKNER, *J. prakt. Chem.*, **57** (1852), 10). Geomyricite occurs at the Gesterwitz brown-coal deposit.

**Georetinic Acid.**—See *Brücknerellite.*

**"German Vaseline."**—This is compounded by dissolving paraffin or ceresin in colorless petroleum oils. See page 643.

**Gilsonite.**—Gilsonite, or uintaite, is a brilliant black, very brittle variety of asphalt having a marked conchoidal fracture. It decrepitates but fuses easily in a candle flame, and is soluble in carbon disulphide and turpentine. Upon exposure to air it readily breaks down into a brown powder. It is mined in Uinta County, Utah.

Gilsonite has been investigated by DAY, *J. Frank. Inst.*, **140** (1895), 221; *Proc. Am. Phil. Soc.*, **37** (1898), 171; ELDRIDGE, *Ann. Rept. U. S. Geol. Survey*, **17** (1896), i, 915; MAGUIRE, *Mines and Minerals*, **20** (1900), 398; and PINE, *Min. World*, **23** (1906), 610.

**Glance Pitch.**—In the glance pitches, which are very hard and brittle bitumens, the saturated hydrocarbons are reduced to between 6 and

7 per cent. Egyptian glance pitch contains 99.7 per cent. of bitumen soluble in carbon disulphide, 8.5 per cent. of sulphur, and 15 per cent. of ash-free residual coke. See *Manjak.*

**Glessite.**—A resin occurring, like *gedanite*, with succinite on the shores of the Baltic; it has a brown color and a specific gravity of 1.015 to 1.027 (HELM, *Schriften Ges. Danzig*, **5** (1881), 291).

**Gloss-Coal.**—A variety of brown coal, compact, deep black, with conchoidal fracture well developed, possessing a resinous to glossy and metallic luster. It is the hardest and most compact of the lignites; its specific gravity varies from 1.2 to 1.5.

**"Glyco."**—A proprietary name for liquid petrolatum, *q.v.*

**Glycol.**—Glycol can be made from oil gas by first chlorinating and then replacing the chlorine atoms by hydroxyl groups.

**"Glycolin."**—A proprietary name for liquid petrolatum, *q.v.*

**"Glymol."**—A proprietary name for liquid petrolatum, *q.v.*

**"Goudron."**—A Russian petroleum product possessing a specific gravity of not below 0.935 and a flash-point of not below 270°C.

**Grahamite.**—A jet-black lustrous hydrocarbon, soluble in carbon disulphide and chloroform, but not in alcohol; it is fusible, has a specific gravity of 1.145, and has a conchoidal fracture. It occurs in vein-like masses in Colorado, Oklahoma, and West Virginia.

Grahamite has been described by BLAKE, *Trans. Am. Inst. Min. Eng.*, **18** (1890), 563; DUMBLE, *ibid.*, **21** (1894), 601; KIMBALL, *Am. J. Sci.*, (3), **12** (1876), 277; RICHARDSON, *J. Am. Chem. Soc.*, **32** (1910), 1032; WHITE, *Bull. Geol. Soc. Am.*, **10** (1899), 277; and WURTZ, *Proc. Am. Assn.*, **18** (1869), 124.

**Grease.**—Properly speaking, this term should only be applied to fatty or oily matter of animal origin; but mixtures of mineral oil with lime- and soda-soaps constitute well-known lubricating greases.

Engine-waste grease is fatty or other lubricating material recovered from cotton-waste or wipe-rags; it has been used in making axle grease and other coarse lubricants.

On lubricating greases, see GILLETT, *J. Ind. Eng. Chem.*, **1**, 351.

**"Green Oil."**—In the Scottish shale-oil industry, the once-run crude oil after chemical treatment. It is distilled in the first-stage oil stills and is fractioned into naphtha, light oil, heavy oil, and heavy oil and wax. See page 835.

**"Green-Tar."**—Barbados petroleum (GODING, *Proc. Sci. Assn. Trinidad*, **1** (1867), 37).

**Grey Shale.**—The grey shale, which was worked at Addiewell, Scotland, lies above the Houston coal, which consists of several thin coal-seams. See page 812.

**Gulf Binder.**—This blown oil is produced from Texas petroleum; it contains 99.4 per cent. of bitumen soluble in carbon disulphide, 70.1 per cent. of bitumen soluble in 88°Bé. naphtha, 48.4 per cent. of pure bitu-

men as saturated hydrocarbons, 0.2 per cent. of paraffin scale, 0.71 per cent. of sulphur, and 14.6 per cent. of ash-free residual coke.

**Gum Resin.**—The semi-solid to solid and usually light colored emulsion or mixtures of true resins with various gums (carbohydrates), constituting the sap of certain plants and trees, partly soluble in water.

**Guyaquillite.**—A pale yellow, amorphous, non-resinous oxygenated hydrocarbon, from near Guyaquil, South America; it has the specific gravity 1.092, begins to fuse at 70°C., and is soluble in alcohol. See JOHNSTON, *Phil. Mag.*, (2), **13** (1838), 22 and 329.

**Hammer Oil.**—Examples of oils used in the lubrication of steam hammers are steam-refined cylinder stocks (*q.v.*), free from tar, green in color, and having a gravity of 25° or 26°Bé., a flash-point of 590° or 610°, a fire test of 680° or 710°, a cold test of 30°, and a viscosity of 220 or 245, Saybolt.

**Hard Pitch.**—Pitch showing a penetration of not more than 10.

**Harness Oil.**—The leather dressing termed "black harness oil" is thus compounded:

| | 1 | 2 |
|---|---|---|
| Leather oil (*q.v.*) | 40 gal. | 40 gal. |
| Oleic acid | 2 gal. | ..... |
| Yellow petrolatum | 12 lb. | 8 lb. |
| Aniline color | 1½ lb. | 1½ lb. |
| Nitrobenzene | 2 parts | 1½ parts |
| Ebonite black wax | ..... | 12 lb. |

The term "harness oil" is also applied to cylinder stock with 5 per cent. of neatsfoot oil. Harness oil should possess a glossy black color.

**Hartine.**—A white resin separated by ethyl ether from a resin from the brown coal of Oberhart (SCHRÖTTER, *Ann. phys. chem.*, **59** (1843), 45). See *Xyloretinite*.

**Hartite.**—This mineral resembles *fichtelite* closely, but melts at 75°C.; it occurs near Gloggnitz, Austria, and in Styria and Carinthia.

HAIDINGER described hartite in *Ann. phys. chem.*, **130** (1841), 261. See also RUMPF, *Sitz. k. Akad. Wiss. Wien*, **60** (1869), 91.

**Harvester Oil.**—(1) A product of high fire test and viscosity. It is sometimes compounded with graphite for use on heavy engines and farm machinery.

(2) Castor machine oil (*q.v.*) is also used on harvester and farm machinery.

**Hatchettine.**—A synonym for hatchettite, employed by CONYBEARE, *Ann. Phil.*, **1** (1822), 136; and JOHNSTON, *Phil. Mag.*, **12** (1838), 338.

**Hatchettite.**—A yellowish white, wax-yellow or greenish yellow hydrocarbon which darkens on exposure; it melts at 46°C., is sparingly soluble in boiling alcohol and cold ether, and is decomposed by concentrated sulphuric acid. Its specific gravity varies from 0.892 to 0.983.

On hatchettite, see DEWALQUE, *Ann. Soc. Géol. Belg.*, **10** (1883), 71; LOHEST, *ibid.*, **10** (1883), 113; CESARO, *ibid.*, **18** (1891), 88; CHANDELON, *Bull. Acad. Roy. Belg.*, **5** (1838), 673; CONYBEARE, *Ann. Phil.*, **21** (1823), 124; HAIDINGER, *Sits. k. Akad. Wiss. Wien*, **2** (1849), 312; MELION, *Lotos*, **5** (1855), 122; and WAGNER, *N. Jahrb.*, 1864, 687.

**Headlight Oil.**—An illuminating oil, suitable for use in the headlight lamps of locomotives; it is usually of 150°F. fire test.

**"Heavy Petroleum Oil."**—A name for liquid petrolatum, *q.v.*

**Helenite.**—A wax near ozokerite, but elastic like caoutchouc; it has a yellow color and a specific gravity of 0.915. It occurs at Ropa in Galicia (NAWRATIL, *Dingler's polyt. J.*, **248** (1883), 513).

**Heneicosane.**—Heneicosane ($C_{21}H_{44}$) has been separated and identified from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565).

**Hentricontane.**—Hentricontane ($C_{31}H_{64}$) has been separated by MABERY (*Proc. Am. Acad.*, **40** (1904), 349) from the solid hydrocarbons that collect in certain Pennsylvania oil wells.

**Heptanaphthylene.**—Concerning heptanaphthylenes, see MARKOVNIKOV, *J. Russ. Phys.-Chem. Soc.*, **34** (1902), 632 and 720; **35** (1903), 1023; **36** (1904), 39; and MARKOVNIKOV and STADNIKOV, *ibid.*, **35** (1903), 389.

**Heptane.**—Heptane has been found in the lighter distillates from the oil of Zarskije Kolodzy, Tiflis (BEILSTEIN and KURBATOW, *Ber.*, **14** (1881), 1620). Normal and iso-heptanes have been isolated from Pennsylvania petroleum. WARREN separated heptane in considerable amount by working on large quantities of Pennsylvania crude oil (*Am. J. Sci.*, (2), **45** (1868), 262).

**Hexacosane.**—Hexacosane ($C_{26}H_{54}$) has been separated from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565). MABERY (*ibid.*, **40** (1904), 355) also separated this hydrocarbon during the course of an investigation into the composition of commercial paraffin.

**Hexadecane.**—Hexadecane ($C_{16}H_{34}$) has been isolated from Pennsylvania petroleum; MABERY (*Proc. Am. Acad.*, **37** (1902), 565) described its separation.

**Hexamethylene.**—On hexamethylene from American and Galician petroleum, see FORTEY, *J. Chem. Soc.*, **73** (1898), 932.

**Hexane.**—Hexane has been found in the lighter distillates from the oil of Zarskije Kolodzy, Tiflis (BEILSTEIN and KURBATOW, *Ber.*, **14** (1881), 1620). Normal and iso-hexanes ($C_6H_{14}$) have been isolated from Pennsylvania petroleum (see WARREN, *Am. J. Sci.*, (2), **45** (1868), 262).

**Highgate Resin.**—See *Copalite*.

**Hircine.**—See *Hircite*.

**Hircite.**—An amorphous, brown, resinoid oxygenated hydrocarbon,

soluble in sulphuric acid and partly so in alcohol (PIDDINGTON, *Arch. Pharm.*, **74**, 318; *J. As. Soc. Beng.*, **10**, 436; **16**, 371).

**Hofmannite.**—A crystalline, colorless, tasteless and odorless mineral, possessing a specific gravity of 1.0565; it melts at 71°C. and has the composition $C_{20}H_{36}O$. It forms a white crystalline efflorescence on lignite in the vicinity of Siena (BECHI, *Trans. Accad. Lincei*, **2** (1878), 135).

**"Honey-Stone."**—See *Mellite.*

**Horn Coal.**—A variety of cannel coal (*q.v.*) from South Wales.

**Huminite.**—A hydrocarbon from Östmark, Sweden, having the composition (ash-free): carbon, 67.15; oxygen, 29.83; hydrogen, 2.55; nitrogen, 0.47; and sulphur, 0.40 per cent. (EKMAN, *Oefv. k. Vet.-Akad. Förh.*, **25** (1868), 138).

**Humus Acid, Native.**—Native humus acid has been found as a black, crumbling coaly mass in the coal-basin at Falkenau, Bohemia (JOHN, *Verhandl. Geol. Reichs.*, Feb. 3, **1891**).

**Hydramyl.**—See *Amyl Hydride.*

**Hydraulic Press Oil.**—The non-viscous neutral oil described under *concrete form oil* has been recommended for use on hydraulic presses.

**"Hydrocarbon."**—Oil of low specific gravity obtained during the compression of oil-gas and sold as petroleum ether (*q.v.*).

**Hydrocarbon Black.**—A name for *lampblack.*

**Hydroline "(B)."**—This blown oil originated in Texas petroleum; it contained 99.8 per cent. of bitumen soluble in carbon disulphide, 68 to 71 per cent. of bitumen soluble in 88°Bé. naphtha, 57 to 64 per cent. of pure bitumen as saturated hydrocarbons, 1 per cent. of paraffin scale, 0.41 to 0.56 per cent. of sulphur, and 8.7 to 12.2 per cent. of ash-free residual coke. It has been reported that "Hydroline '*B*'" was the same as *byerlyte.*

**Ice-Machine Oil.**—An oil of low cold-test, as, for example, the light lubricating oils from Texas crudes. Light spindle oil of 26° to 27°Bé. gravity, 60 to 100 sec. Saybolt viscosity at 70°F., 325° to 360°F. flash, and 0° to 4°F. cold test, has also been recommended for refrigerating machines.

**"Ichthynat."**—A preparation of a similar character to ichthyol (*q.v.*), introduced for medicinal purposes. On its composition, see PASSMORE, *Chem. and Drug.*, **75** (1909), 935.

**Ichthyol.**—When the bituminous shales containing fossil fish found at Seefeld in the Tyrol, are dry-distilled, a crude volatile oil is obtained, which, when treated with an excess of concentrated sulphuric acid and subsequently neutralized with ammonia, yields the product termed "ichthyol," the ammonium salt of ichthyol sulphonic acid. It is used in ointments as a stimulant.

On the composition of ichthyol, see PASSMORE, *Chem. and Drug.*, **75** (1909), 935; and BAUMANN and SCHOTTEN, *Pharm. Zentralh.*, **1883**, 447.

**"Ichtosan."**—See comments regarding "ichthynat."

**Idrialine.**—See *Idryl.*

**Idrialite.**—A white mineral, crystalline when pure, which fuses at 205°C.; it occurs in Idria. GOLDSCHMIEDT (*Sits. k. Ak. Wiss. Wien*, 80 (1880), 290) has investigated a pistachio-green mineral resin of Idria, occurring in nodules and as an incrustation at the mercury mines; this is also termed "idrialite."

On idrialite, see SCHARIZER, *Verh. k.-k. geol. Reichs.*, 1881, 335; DUMAS, *Ann. chim. phys.*, 50 (1832), 360; and SCHRÖTTER, *Z. phys. math.*, 3, 245; 4, 5. See *Idryl.*

**Idryl.**—BÖDEKER reported that a black material obtained from the mercury condensation-chambers at Idria afforded a substance which he termed *idryl*, supposing it to be the radical of *idrialite*.

On idryl, see BÖDEKER, *Ann.*, 52 (1844), 100; GOLDSCHMIEDT, *Sits. k. Akad. Wiss. Wien*, 76 (1878), 319; 81 (1880), 415; and SCHRÖTTER, *Ber. deutsch. Nat. Versamml.*, 1887, 117.

**Illuminating Oils.**—These oils are the products heavier than gasoline, ranging from 90° to 250°F. flash-point. Like gasoline, they are divided into two general classes, depending whether they are normal or "cracked" products. The usual tests applied to burning oils are: gravity, flash-point, fire test, distillation, sulphur determination, viscosity, color and burning tests.

There are several grades of illuminating oils, which may be roughly classified as follows: 1. 45° to 47°Bé. gravity, usually called "water-white" burning oil (normal product); fire test, 150°F. or better. 2. 43° to 45°Bé. gravity, usually called "standard white" burning oil ("cracked" product); fire test, 100°F. or better. 3. Below 43°Bé. gravity, a cheaper product which may be either normal or "cracked;" fire test, 110°F. or better. 4. 300°F. fire test, known as "300° Oil," "Mineral Sperm," "Mineral Colza" or "Mineral Seal."

The first three grades are ordinary kerosene lamp oils. "300° Oil" is used as an illuminating oil in cases where a great degree of safety is required.

The most practical test to apply to illuminating oils is to make an actual burning test, taking into consideration the incrustation of the wick and diminution of the flame. These tests are merely comparable and require some practice in order to judge the quality of the oil. Provided the flash is satisfactory, the best burning oil is that with the lowest viscosity, lowest iodine absorption, highest gravity, lowest sulphur, and best color.

See *Kerosene.*

**Impsonite.**—An asphalt found in Oklahoma; it is much like albertite (*q.v.*), but is almost insoluble in turpentine.

**Incubator Oil.**—An illuminating oil possessing a gravity of 48° and a fire test of 150°F. Such an oil should be low in cold test, and burn a

lamp out dry with as little as possible diminution of flame or charring of wick.

**Insect Oil.**—A non-viscous neutral oil of the same grade as *concrete form oil* or *paint oil.*

**Intermediate Oils.**—The specific gravity of these distillates from shale oil varies from 0.84 to 0.87; they are used for gas-making and at 800° to 900°C. a good Scottish intermediate oil will yield 1,200 c.p. per gallon.

See *Gas Oil.*

**"Interol."**—A proprietary name for liquid petrolatum, *q.v.*

**Ionite.**—A fossil hydrocarbon found in a more or less impure condition in the lignite of Ione Valley, Amador Co., Cal.; it has a brownish yellow color and, while only partially soluble in alcohol, is completely dissolved by chloroform; it yields a brown tarry oil on destructive distillation (PURNELL, *Min. Sci. Press*, Mar. 24, **1877**; *Am. J. Sci.*, **16** (1878), 153).

**Isodurol Derivatives.**—These are considered by BIELEFELDT in *Ann.*, **198** (1879), 380.

**"Isurol."**—See comments regarding "ichthynat."

**Ixolyte.**—An amorphous, hyacinth-red, greasy hydrocarbon mineral which softens at 76°C. and resembles *hartite*; it is found at Oberhart, near Gloggnitz, Austria. Ixolyte was described by HAIDINGER in *Ann. phys. chem.*, **132** (1842), 345.

**Jaulingite.**—A resin found in the Jauling, near St. Viet, Lower Austria; it resembles *succinite* (see ZEPHAROVICH, *Ber. Akad. Wiss. Wien*, **16** (1855), 366).

**Jelly, Mineral.**—See *Petrolatum.*

**Jet.**—Jet, azabache, gagat and black amber are classes of lignite. The name "gagat" is derived from the river Gages, in Lycia, at whose mouth this substance occurs. A dense, jet-like coal, with conchoidal fracture, so hard and firm that it can be filed and polished for ornaments. The Bentheim gagat is a vein in sandstone, not a stratum.

See *Lignite.*

**"Jet Black."**—A name for *lampblack.*

**Journal Oil.**—See *Machine Oil.*

**Jura Asphalt.**—On the origin of Jura asphalt, see SCHARDT, *Arch. sci. phys. Genève Nat.*, (4), **23** (1907), 611.

**Kabaite.**—A hydrocarbon related to ozokerite or scheererite, found in meteorites (MEUNIER, *Compt. rend.*, **109** (1889), 977).

**Kaiser Oil.**—A burning oil of European manufacture; it possesses a specific gravity of 0.780 to 0.800 at 15°C. See BUCHNER, *Dingler's polyt. J.*, **227** (1878), 381.

**Kandol.**—See *Canadol.*

**Kerogen.**—A term applied to the bituminous material in Scottish oil-shale; upon destructive distillation of the shale, kerogen gives rise to crude shale oil. See pages 807 and 816.

**Kerosolene** (*Kerosoline*).—A mixture of hydrocarbons from coal or albertite oil, equivalent to petroleum ether, possessing a boiling point of 90°F.; it was proposed by BIGELOW (*Boston Med. Surg. J.*, July 11, 1861) as an agent for producing anesthesia by inhalation. On kerosolene, see also PARISH, *Am. J. Pharm.*, (3), 9 (1861), 396.

**Kerosene.**—A mixture of hydrocarbons whose average boiling point is about 450°F., freed on the one hand from gasoline or naphtha and on the other hand from the heavy hydrocarbons that belong to gas oil and lubricating oil, which would make the oil act badly in lamps. Kerosene contains nonane ($C_9H_{20}$), decane ($C_{10}H_{22}$), etc., and is used as an illuminating oil. The following are the rules of the New York Produce Exchange relating to kerosene: "Refined petroleum shall be standard white or better, with a burning test of 110°F. or upward, and of a gravity not below 44°Bé., 'United States Dispensatory' standard.

"The burning test of refined petroleum shall be determined by the use of the Saybolt electric instrument, and shall be operated in arriving at a result as follows: In 110° and upward, the flashing-points, after the first flash (which will generally occur between 90° and 95°), shall be taken at 95°, 100°, 104°, 108°, 110°, 112° and 115°. In 120° and upward, after first flash, at 100°, 105°, 110°, 115°, 118°, 120°, 122°, and 125°. In 130° and upward, every 5° until burning point is reached."

Two well-known kerosenes from Pennsylvania petroleum possess respectively gravities of 48.5° and 45° and fire tests of 150°F. and 175°F. See *Illuminating Oils.*

Generally speaking, the burning oils manufactured in the United States are of the following grades: Fire test, 110°F.; Abel test, 70°F. Fire test, 120°F.; Abel test, 73°F. Fire test, 150°F. The last-mentioned is nominally "water-white," and the others range in color from "prime-white" to "standard-white" (straw color to pale yellow).

**Kerosene Distillate.**—(1) See "*Kerosene Stock.*" (2) See *Power Distillate.*

**Kerosene Shale.**—Speaking broadly, this term may be applied to any bituminous or oil shale from which illuminating oil has been or may be obtained. The term is specifically applied, however, to the oil-shale found in New South Wales, although this kerosene shale is more of the nature of a cannel coal or a torbanite, and does not possess the properties of a shale. The best grades of kerosene shale yield 150 gal. of crude oil per ton, but the shale at present being worked by two companies of New South Wales yields from 60 to 120 gal. of oil. See page 842.

See *J. Roy. Soc. Arts*, **56**, 748; BAKER, *U. S. Cons. Rept.*, **1908**, No. 333, 107; CARNE, *Mem. Geol. Survey N. S. Wales, Geol.*, No. 3 (1903), 333; DAWKINS, *Rept. Brit. Assn.*, **1886**, 643; PLUMMER, *Eng. Min. J.*, **78** (1904), 66; ROBERTSON, *Trans. Min. Inst. Scot.*, **14** (1892), 88; SKEY, *Ann. Rept. Col. Mus. Lab. N. Z.*, **23**, 50; **25**, 56; **29**, 19; and **31**, 10.

See *Shale Oil.*

**"Kerosene Stock."**—(1) "Burning oil stock," or "burning oil distillate," described on page 460. (2) In California practice, a product having an amber color and a gravity of 40° to 42°Bé.

**Kerosenic Acid.**—The preparation of pure kerosenic acid and its salts has been described by TIDEMAN in *Trudi Bak. Otd. Imp. Russk. Techn. Obsch.*, **1893**, 56.

**Kimmeridge Shale.**—Extensive deposits of bluish gray slaty clay, containing more or less volatile matter, and interstratified with thin beds of highly bituminous shale, occur in Dorsetshire. This clay, which is a member of the Upper Oolite, attains in places a thickness of as much as 600 ft. The deposits, which take their name from the village of Kimmeridge, in the Isle of Purbeck, were worked as early as the sixteenth century, as a source of alum. Locally the shale is known as "Kim-coal," and it has been used in the district as an inferior fuel.

Of the various seams, the richest in bituminous matters appears to be that which is termed the Blackstone. This bed is about 2 ft. in thickness, and in laboratory experiments the material gave 39 per cent. of oily and solid volatile products (2.3 per cent. of light oil or naphtha, and 36.7 per cent. of heavy oil containing 1.9 per cent. of paraffin), 18 per cent. of gas, water, ammonia, etc., and 43 per cent. of coke. In 1876, Patterson, of the Warrington Gas Works, reported that a ton of the Kimmeridge shale yielded an average of 67.25 gal. of crude oil (containing 20 per cent. of naphtha and 3 per cent. of paraffin), 11 gal. of liquor containing 6 per cent. of ammonia, and from 1118 to 1187 lb. of coke.

See BURTON GREEN'S "Kimmeridge Shale; its Origin, History, and Uses," London, **1886**; and MANFIELD, *Min. J.*, Feb. 19, **1916.**

**"Kingle."**—Barren "blaes," or ribs of hard calcareous or quartzose matter, destitute of bituminous matter, occurring in the Scottish oil-shales.

**"Kiton."**—A mixture of clay and tar, proposed by RASCHIG for dust-laying.

**Knitting Machine Oil.**—The following neutral oils have been recommended for knitting machines lubrication:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 32¾°Bé. | 415°F. | 480°F | 20°F. | 200 |
| 34° | 340° | 400° | 20° | 80 |
| 34½° | 340° | 400° | 20° | 75 |
| 35° | 340° | 400° | 20° | 70 |

**Köflachite.**—A resin occurring in the Tertiary brown coal of Laukowitz near Köflach in Styria; it has a specific gravity of 1.2 to 1.25, fuses

at 98°C., is soluble in ether and carbon disulphide, and is reported to have the composition $C_{20}H_{40}O_2$ (Doelter, *Mitth. Ver. Steiermark*, 1878, 93).

**Künlite.**—A reddish brown to yellow, soft, amorphous hydrocarbon mineral which has a specific gravity of 0.88, a melting point of 114°C., and distils at 200°C.; it is found at Utznach, Switzerland, and at Redwitz, Bavaria (Kraus, *Ann. phys. chem.*, 43 (1838), 141; Trommsdorff, *Ann.*, 21, 126).

**Krantzite.**—A sectile, somewhat elastic yellow resin, which possesses a specific gravity of 0.968, fuses at 225°C., and whose composition corresponds nearly to the formula $C_{40}H_{64}O_4$ (Bergemann, *J. prakt. Chem.*, 76 (1859), 65).

**Lake Oil.**—A Russian fuel oil which has a specific gravity of 0.895 to 0.935 and a flash-point of not below 70°C.

**"Lake Pitch."**—Asphalt from the Pitch Lake, Trinidad; lake pitch is richer than the land pitch (*q.v.*) in bituminous matter soluble in petroleum spirit.

**Lampblack.**—A product obtained directly from natural gas by burning the latter under plates or rolls. On the manufacture of lampblack, see Cabot, *J. Soc. Chem. Ind.*, 13 (1894), 128; Butler, *Min. Eng. World*, Oct. 28, 1911; and Neff, *Dingler's polyt. J.*, 231 (1879), 177.

**Lamp Oil.**—See *Illuminating Oils* and *Kerosene.*

**"Land Pitch."**—Asphalt from the deposit in Trinidad lying between the Pitch Lake and the seacoast.

See *"Lake Pitch."*

**Leaf-Coal.**—Leaf-coal, or foliated coal, is a lignite formed of superimposed layers; Westerwald leaf-coal contains 62.8 per cent. of carbon, 6.7 per cent. of hydrogen, 17.3 per cent. of oxygen and nitrogen, 11 per cent. of ash and 24.6 per cent. of water.

**Leaf-Shale.**—The products of the distillation of leaf-shale are as follows:

| | |
|---|---|
| Tar: specific gravity, 0.88 to 0.93 | 5 to 25 per cent. |
| Crude distillate: specific gravity, 0.70 to 0.85 | 0.10 to 6 per cent. |
| Crude distillate: specific gravity, 0.85 to 0.90 | 3 to 12 per cent. |
| Crude paraffin | 0.25 to 1 per cent. |

(Wagenmann, *Dingler's polyt. J.*, 145 (1857), 309).

**Leather Oil.**—A non-viscous neutral oil of the same grade as *concrete form oil.*

**Leucopetrin.**—See *Leucopetrite.*

**Leucopetrite.**—A substance, between a resin and wax in character, found in a brown coal at Gesterwitz, near Weissenfels; it crystallizes in white needles from ether and boiling absolute alcohol, and melts above 100°C. According to Brückner (*J. prakt. Chem.*, 57 (1852), 1), it has the formula $C_{30}H_{64}O_3$.

**Libollite.**—A kind of asphalt occurring near Libollo, in western Africa; it resembles *albertite* (GOMES, *Comm. Dir. Trabalhos Geol. Portugal*, **3** (1896–8), 244 and 290; **4**, 206).

**Light Ligroin.**—See *Canadol.*

**Lighthouse Oil.**—The United States Lighthouse establishment requires that mineral oil supplied for use in lighthouse lamps shall possess a specific gravity of not less than 0.802, a flash-point of not less than 140°F., and a fire test of not less than 154°F. In a fifth-order Hains lamp, the light is to be equal to that of eighteen sperm candles and is to continue undiminished during 5 hr. burning.

**"Light Petroleum."**—A petroleum product possessing a boiling point of 70°–90°C. and a specific gravity of 0.650–0.666 (85°–80°Bé.). It is employed as a technical solvent for caoutchouc, asphalt and fatty oils, and for the carburation of air in gas machines. The light petroleum used in the laboratory for extractions varies from 88°Bé. to 62°Bé.

**Light Slushing Oil.**—A non-viscous neutral oil of the same grade as *concrete form oil.*

**Lignite.**—Under this name are comprised a number of substances which lie between peat on one side and bituminous coal on the other.

The methods in use in the destructive distillation of brown coal, or lignite, an industry of some importance in Saxony, bear a close resemblance to those on which the shale oil industry of Scotland is founded. The tar obtained from lignite by slow distillation, is fractionally distilled and purified. For information relating to the treatment and composition of lignite tar, see VON BOYEN, *Chem.-Ztg.*, **14**, 267 and 289; *Z. angew. Chem.*, **1891**, 101 and 261; and HEUSLER, *Ber.*, **25** (1892), 60 and 65.

For the chemistry of lignite, see CLARKE, *Bull. U. S. Geol. Survey*, No. **491**, 711. On methods of utilizing lignite, see DARLING, *J. Gas Ltg.*, **131** (1915), 456.

See *Pitch-Coal.*

**Lignite Oils.**—Heavy lignite oils possess a specific gravity of 0.89 to 0.97, a faint odor of creosote, and usually a low viscosity. They dissolve to the extent of 20 to 60 per cent. when shaken with two volumes of cold ethyl alcohol.

**Ligroin.**—A term rather loosely applied. It may denote a refined petroleum distillate of boiling point 120°–135°C., but is occasionally applied to lower boiling fractions, such as benzoline; in general, it is held to be another name for *light petroleum* (*q.v.*). Russian "ligroine" is a naphtha having a boiling point of 100° to 120°C. and a specific gravity of 0.707 to 0.722 at 15°C.

On the distillation of ligroin, see REICHARDT, *Apoth.-Ztg.*, **1888**, 12.

**Limmer Asphalt.**—Limmer rock contains 8.26 per cent. of material soluble in carbon disulphide. It has been described by HOFFMANN, *Z. prakt. Geol.*, **3** (1895), 370.

**Linseed Oil Adulterant.**—See *Paint Oil.*

**"Liquid Albolene."**—A proprietary name for liquid petrolatum, *q.v.*

**Liquid Bituminous Material.**—Bituminous material which shows a penetration at normal temperature under a load of 50 grams applied for 1 sec. of more than 350.

**Liquid Fuels.**—The liquid fuels most frequently used are: petroleum and refinery residues, shale oil, tar from the dry distillation of coal and wood, the tar oils obtained by the distillation of coal tar, benzine, and denatured, methylated, or wood alcohol.

**On liquid fuels, see Booth's "Liquid Fuel and Its Apparatus," New York, 1912.**

**Liquid Paraffin.—A name for liquid petrolatum, *q.v.***

**Liquid Petrolatum.—See *Petrolatum* (*Liquid*).**

**"Liquid Saxoline."—A proprietary name for liquid petrolatum, *q.v.***

**"Litho-Carbon."—A product obtained from bituminous limestone of Uvalde County, Texas. It has been used in varnish making and for insulating purposes.**

**"Litholine."—A registered name for petrolatum, *q.v.***

**Loom Oil.—A heavy spindle oil. The following viscous neutral oil is employed for heavy looms: gravity, 31°Bé.; flash-point, 415°F.; fire test, 480°F.; cold test, 20°F.; and Saybolt viscosity, 210. A non-viscous** neutral oil possessing a gravity of 34.5°Bé., a flash-point of 340°F., a fire test of 400°F., a cold test of 20°F., and a Saybolt viscosity of 75, is, however, sometimes used on looms when a light oil is desired.

**Lubricating Greases.**—These lubricants, which are employed to some extent on account of their easy and, in some cases, economical application, consist of solutions of lime-soaps or lime-alkali-soaps in mineral oils; mixtures of wool-grease, tallow, alkali soaps, etc., with mineral oil; or, in the case of axle or wagon greases, of lime-soaps, rosin oil, lignite-tar, coal-tar oils, magnesium silicates (talcum), etc. See "*Oildag.*"

**Lubricating Oil.**—See *Cylinder Stocks, Neutral Oil, Paraffin Oils,* and the various lubricating oils for special purposes, as, for example, *Engine Oil, Machine Oil, Spindle Oil, Transformer Oil,* etc.

**Lyncurium.**—See *Succinite.*

**Machine Oil.**—Three grades of machine oil are made by some refiners from paraffin-base petroleum: these possess gravities of 29.4°, 29°, and 28.5°Bé.; Saybolt viscosities of 295, 400, and 700 at 70°F.; flash-points of 415°, 430°, and 440°F.; fire tests of 480°, 490°, and 500°F.; and cold tests of 20°F., 15°F., and 15°F., respectively. The general color is red. A viscous neutral oil, stock-loaded and red in color, possessing a gravity of 30.5°Bé., a flash-point of 395°F., a fire test of 450°F., a cold test of 30°F., and a Saybolt viscosity of 240, is also marketed under this name.

**Machinery Oils.**—See *Engine Oil, Machine Oil, Neutral Oil, Cylinder Stocks,* and the various lubricating oils which are defined under their trade-names, as *Compressor Oils, Dynamo Oils,* etc.

**Maltha.**—A dark colored and viscous, naturally occurring hydrocarbon complex, insoluble in water, but completely soluble in carbon disulphide, benzol, etc. It is also termed *pissasphalt.*

**Manjak.**—A variety of bitumen found in Uvalde County, Texas (*Smithson. Rept.*, **1889**, 445).

On Barbados manjak, see BEDSON, *Trans. Inst. Min. Eng.*, **16** (1899), 388; EMTAGE, *J. Soc. Arts*, **52** (1904), 367; and MERRIVALE, *Trans. Fed. Inst. Eng.*, **14**, 539; **16** (1898), 33.

On Marbela manjak, see GUPPY, *Geol. Mag.*, **1** (1904), 276.

On Trinidad manjak, see HENRY, *Bull. Imp. Inst.*, **1** (1903), 180; and RASPASS, *Trans. Inst. Min. Eng.*, **36** (1909), 119.

See also *Asphalt.*

**"Marine Oil."**—An illuminating oil for use on ships. A refined distillate between the specific gravities 0.825 and 0.848, with a flash-point of 225°F., has been recommended for marine purposes. Not to be confounded with marine animal oils (fish oils, liver oils, and blubber oils).

**Mastic.**—A mixture of bituminous material and fine mineral matter, suitably made for use in highway construction and for application in a heated condition.

"Mastic" is prepared for use in paving by mixing hot asphalt-rock, ground to a fine powder, with such proportion of hot bitumen, similar to that contained in the natural rock, that the product contains about 15 per cent. of bitumen and 85 per cent. of limestone. "Gritted asphalt-mastic" is composed of the mastic already described, remelted with 5 per cent. of bitumen, and from 30 to 40 per cent. of clean dry fragments of limestone or sand. For use in preparing mastic, the bitumen extracted from Seyssel asphalt is stated to possess the desired qualities in the highest degree.

See LEVY, *Bull. soc. ind. min.*, (2), **1** (1872), 67.

**Masut.**—A Russian petroleum product: crude oil deprived of volatile light substances by exposure to air—specific gravity above 0.880; flash-point above 70°C. (158°F.).

See *Ostatki.*

**Mat.**—See *"Carpet."*

**Matrix.**—A composition or material forming a cushion, or binding the aggregate together, for use in the construction of pavements.

**Mazout or Mazut.**—See *Masut.*

**Medicinal Oil.**—See *Petrolatum* (*Liquid*).

**"Med-O-Lin."**—A proprietary name for liquid petrolatum, *q.v.*

**Melanasphalt.**—On this variety of asphalt, see WETHERILL, *Trans. Am. Phil. Soc.*, (2), **10** (1853), 353.

**Melanchyme.**—A bituminous substance found in masses in the brown coal of Zweifelsruth, Bohemia (HAIDINGER, *Lotos*, **1**, 85, 216; **6**, 86; **8**, 3). That part of this substance which was soluble in alcohol was termed *rochlederite*, the residue *melanellite.*

**Melanellite.**—That portion of *melanchyme* which is insoluble in alcohol; it is black and gelatinous.

**Mellite.**—A rare mineral, also called "honey-stone," which occurs

in tetragonal crystals and nodular masses of a honey-yellow color in the Thuringian lignite beds; it is said to be an aluminum mellitate.

See MELION, *Lotos*, 5 (1855), 122.

**Menat Oil-Shales.**—For accounts of these shales, see BOURIOT, *Bull. Soc. Géol. France*, (2), 8 (1850), 39; and LECOQ, *Ann. Sci. Ind. Auvergne*, 2 (1829), 433.

**Mersey "Yellow Coal."**—See *Tasmanite*.

**Mesitylene.**—Mesitylene, an isomer of cumene, was isolated by LACHOWICZ from Galician oil, by ENGLER from the petroleums of America, Hanover, Galicia, Italy, and Elsass, and by MARKOVNIKOV from the oil of Baku. PONI (*Ann. Sci. Univ. Jassy*, 2 (1903), 65) reported the presence of mesitylene in Rumanian oil, in the portion boiling between 100° and 200°C. KRÄMER and BÖTTCHER found mesitylene in German petroleum.

On the presence of mesitylene in petroleum, see ENGLER, *Ber.*, 18 (1885), 2234.

**"Meteor".**—A Russian illuminating oil which has a specific gravity of 0.806 to 0.810 and a flash-point not below 28°C. by the Abel-Pensky test.

**Meter Oil.**—An oil of low cold test, like the light lubricating oils from Texas crudes. See *Ice-Machine Oil*.

**Methylpentamethylene.**—On the presence of this hydrocarbon in Caucasian petroleum ether, see ASCHAN, *Ber.*, 31 (1898), 1803.

**Mexican Fluxes.**—The flux produced from Mexican petroleum is differentiated from paraffin fluxes, semi-asphaltic fluxes and asphaltic fluxes by marked characteristics, such as the fact that it contains a high percentage of sulphur, yields a high ash-free residual coke, contains 2 per cent. or more of hard paraffin scale, while only 80 per cent. of it is soluble in 88°Bé. naphtha, as compared to over 90 per cent. in the case of the other residuals. However, as far as the density and percentage of saturated hydrocarbons are concerned, Mexican fluxes can be regarded as asphaltic fluxes. See *Flux*.

**Middletonite.**—A brown, resinous, brittle mineral found between layers of coal at the Middleton collieries, near Leeds, England, and also at Newcastle; it had the specific gravity 1.6, was not altered at 210°C., and was soluble in cold concentrated sulphuric acid.

See EMBLETON, *Trans. Midland Inst. Eng.*, 6 (1878), 154; and JOHNSTON, *Phil. Mag.*, (2), 12 (1838), 261.

**Mine Machine Oil.**—A non-viscous neutral oil of the same grade as *concrete form oil* or of black lubricating oil of −15°F. cold test.

**Mineral Adipocire.**—See *Hatchettite*.

**Mineral Burning Oils.**—Oils suitable for burning with a wick, *e.g.*, belmontine oil, cazeline oil, colzarine oil, mineral seal oil, and pyronaphtha.

See *Illuminating Oils* and *Kerosene*.

**Mineral Caoutchouc.**—See *Elaterite* and *Helenite*, also *Caoutchouc*.

**Mineral Charcoal.**—A fibrous, soft, charcoal-like substance, found covering the surfaces between layers of coal; it has been observed in coal of all ages.

**"Mineral Colza," or "Mineral Colza Oil."**—See *"Mineral Seal Oil."*

**"Mineral Gelatine."**—A base for castor machine oils, *q.v.*

**"Mineral Glycerine."**—A name for liquid petrolatum, *q.v.*

**"Mineral Jelly."**—See *Petrolatum.*

**"Mineral Naphtha."**—See *Naphtha.*

**"Mineral Oil."**—A name applied to liquid petrolatum, *q.v.*

Also a name for both crude petroleum and the lubricating oils produced therefrom.

**Mineral Pitch.**—See *Asphalt.*

**Mineral Resins.**—A term applied to the solid bitumens (see *Bitumen*).

**"Mineral Seal Oil."**—A term which is applied to an oil of the gravity 38.5° to 39°Bé., adapted for lighthouse and locomotive lights. It has a fire test of 300°F., a flash-point of 255°F., and a viscosity of 45 to 50 at 100°F. on the Saybolt universal instrument.

**Mineral Sperm Oil.**—See *"Mineral Seal Oil."*

**"Mineral Turpentine."**—See *Turpentine Substitutes.*

**Miners' Oil.**—This term is usually applied to a neutral oil of the same grade as *paint oil.*

66° to 68°Bé. gasoline is used in safety gasoline lamps; sperm oil or a mixture of 60 per cent. of sperm oil and 40 per cent. of *mineral seal oil* is employed in safety oil lamps.

"Compounded miners' oil" may consist of the grade described as *paint oil* or as *mineral seal oil*, compounded with either cottonseed oil or refined white fish oil. Such oil must possess a good cold test.

**"Miners' Wax."**—A refined paraffin wax of the melting point 118° to 120°F.

**"Min-Seed Oil."**—A bloomless petroleum product, used in connection with linseed oil for cheapening purposes.

See *Paint Oil.*

**"Mixing Oils."**—"Mixing oils" are prepared in Russia from solar oil distillate, the fractions of a lower specific gravity than 0.860 being rejected, and only those possessing a gravity between that figure and 0.885 being employed. The principal use of such oils is said to be in the manufacture of artificial olive oil.

**Montan Wax.**—This wax, used principally as a carnauba wax substitute in the manufacture of polishes and as an insulating material in place of ceresin, was first prepared by VON BOYEN (German Patents 101373 and 116453; *Z. angew. Chem.*, **14** (1901), 1110) from the bitumen extracted from dried Thuringian lignite by means of volatile solvents or from freshly mined lignite by treatment with superheated steam.

On montan wax, see VON BOYEN, *loc. cit.*; EISENREICH, *Chem. Rev. Fett- Harz-Ind.*, **16** (1909), 211; LUDECKE, *Seifenseid.-Ztg.*, **34** (1907),

1065, 1113, 1133, 1153; **37** (1910), 291, 335, and 383; and Schmidt, "Handwörterbuch Schweiz. Volkswirtsch.," **3** (1907).

**Montanin Wax.**—A product resembling Montan wax (*q.v.*), but differing in its physical properties. See *Proc. Roy. Dublin Soc.*, **12** (1909), 202.

**Moor-Coal.**—Moor-coal shows no wood-like structure, and has a flat, conchoidal fracture. It is usually soft and friable, but is occasionally hard and slate-like. In color it varies between dark brown and pitch black, and has a dull appearance. Its specific gravity is 1.2 to 1.3; its hardness, 2.5. It frequently contains remains of marsh-plants. Moor-coal generally accompanies *lignite*, and is found under and interbedded with it.

**Mosquito Oil.**—See *Insect Oil.*

**Motor Gasoline.**—Gasoline of either 73° to 76°Bé. or 66° to 68°Bé. gravity is recommended for motors. Sometimes, however, either of these is mixed with deodorised naphtha possessing a gravity of 58° to 60°Bé. See *Gasoline.*

The properties of certain German motor oils follow:

| Product | Density | B.p., C. | Setting point, C. | Use |
|---|---|---|---|---|
| Galician gas oil | 0.868 | 300°–380° | +1 | Diesel motors. |
| American gas oil | 0.858 | 300°–380° | −10 | Diesel motors. |
| Motor benzine | 0.705/15 | 60°–120° | −20 | Automobile and small motors. |
| "Veloxin" | 0.69/70 | 50°–110° | −20 | Automobile and small motors. |
| Motor benzine II | 0.75/76 | 90°–160° | −20 | Automobile and small motors. |
| Automobile benzine | 0.69/70 | 60°–100° | ..... | Automobile and small motors. |
| Motor benzine I | 0.715/25 | 70°–120° | ..... | Automobile and small motors. |
| Motor benzine II | 0.75/77 | 100°–140° | ..... | Automobile and small motors. |
| Automobile benzine | 0.692/98 | 40°–110° | −70 | Automobile and small motors. |
| Motor benzine I | 0.704/15 | 40°–120° | −70 | Automobile and small motors. |
| Motor benzine "Heraklin" | 0.734/74 | 75°–135° | −70 | Automobile and small motors. |
| Motor benzine II | 0.751/58 | 70°–150° | −40 | Automobile and small motors. |
| Gas oil | 0.864/88 | 174°–360° | 0 | Diesel motors. |
| Gas oil I | 0.863 | 190°–300° | ..... | Diesel motors. |
| Austrian gas oil | 0.860 | ..... | ..... | Diesel motors. |

For a general account of motor fuels, see LEWES, *Am. Gas Light J.*, **103** (1915), 165, 170 and 178. On motor gasoline, see *Technical Paper* **166** of the *Bureau of Mines.*

**Motor Oil.**—A viscous neutral oil, identical with *compressor oil.*

On motor cylinder lubrication, see BRYAN, *J. Am. Soc. Mech. Eng.*, **37**, 293.

**Motor Spirit.**—The saturated aliphatic hydrocarbons of American oil, the polymethylenes from Baku oil or the unsaturated hydrocarbons derived from shale oil, as well as benzene ($C_6H_6$) and alcohol ($C_2H_5.OH$),

are employed in internal combustion engines. The boiling points are usually below 120°. See *Motor Gasoline.*

**Motorcycle Oil.**—This lubricant is usually of the same grade as either *compressor oil* or *aeroplane oil.*

**Mould Oil.**—Glass works mould oil is of the same grade as *compressor oil.*

China works mould oil is of two recommended grades: *transformer oil* and *summer black oil.*

**Mountain Tallow.**—A term used by BRANDE (*Edinb. Phil. J.*, **1824,** 11) for *hatchettite, q.v.*

**Muckite.**—A resin from the coal beds at Neudorf, Moravia; it fuses between 290° and 310°C., has a specific gravity of 1.0025, and a composition corresponding to $C_{20}H_{28}O_2$ (VON SCHRÖCKINGER, *Verh. Geol. Reichs.*, **1878,** 387).

**Mungle Shale.**—The Mungle shale, which averages about 2 ft. in thickness, was formerly worked in the West Calder district, Scotland. It is separated from the Grey shale by the "two-foot coal," which was formerly worked as cannel at Drumcross, near Bathgate. See page 812.

**"Mush."**—A greasy mud, sometimes found on bituminous crusts.

**"Muthol."**—A proprietary name for liquid petrolatum, *q.v.*

**Napalite.**—A peculiar waxy hydrocarbon ($C_6H_4$), found in the Phoenix Mine, near Middletown, Cal.; it has a dark reddish brown color, is brittle, begins to fuse at 42°C., and boils above 300°C. (BECKER, *Monog. U. S. Geol. Surv.*, **13,** 372 (1888)).

**Naphtéine.**—This mineral is described in *Mém. Soc. Agr. Angers*, **2** (1834), 139.

**Naphtha.**—This term, which was formerly applied to any fluid and volatile variety of bitumen, is now generally held to include all those petroleum fractions which boil below 280°F.; but this limit is not observed by all refiners and consequently commercial naphthas may present wide variations. In the valuation of naphtha, stress is laid on a mild odor and a water-white color.

The term naphtha is also applied to the less volatile portion obtained on redistilling benzine and boiling from about 95° to 120°C. The term is unfortunately very loosely used, and is synonymous with "mineral naphtha." The best practice is to confine the designation naphtha to mixtures of light hydrocarbons intended for some purpose that requires a very good odor, such as dry-cleaning, varnish-making, soap-making, etc. Cf. *gasoline.* "Solvent naphtha," however, is not a petroleum product. See page 913.

"Green naphtha" is one of the sondensates obtained in the fractionation of crude shale oil. See page 831.

Benzine, or naphtha, from Pennsylvania petroleum has a gravity of 55° to 63°Bé.

**Naphthalene in Petroleum.**—Although naphthalene, acenaphthene,

fluorene, anthracene, and phenanthrene are found in coal tar, the only one which appears to be *undoubtedly* present in crude petroleum is naphthalene, although it is usually stated that anthracene, chrysene, pyrene, and fluorene are present in small quantity (KRÄMER and BÖTTCHER, *Ber.*, **20** (1887), 595). In this connection, it may be mentioned that BOUSSINGAULT (*Compt. rend.*, **22** (1846), 667; **96** (1883), 1452) mentioned the deposition of naphthalene on cooling the fluid "bitumen" from the burning springs of Ho-Tsing, in the province of Szechuen in China, and that DE LA RUE and MÜLLER (*Proc. Roy. Soc.*, **8** (1857), 221) found naphthalene in Rangoon oil. KRÄMER (*Sitz. Ver. Beförd. Gewerbfl. Preuss.*, **1885**, 299) stated that naphthalene crystallized out from the distillate of 200° to 300°C. from the oil of Tegernsee and Oelheim, but mentioned that this body was not necessarily present in the crude oil.

**Naphthenes.**—Hydrocarbons of the $C_nH_{2n}$ series, which belong, not to the ethylene group, but to what is known as the naphthene, or $C_nH_{2n-6} + H_6$, group, are found in most oils, but especially in that of Baku, and have been examined by BEILSTEIN and KURBATOW (*Ber.*, **13** (1880), 1818, 2028), SCHÜTZENBERGER and JONINE (*Compt. rend.*, **91** (1880), 823), MARKOVNIKOV and OGLOBLIN (*Ann. chim. phys.*, (6), **2** (1884), 372), and others. The naphthenes closely resemble the paraffins. LACHOWICZ ascertained their presence in Galician oil, which he states is about intermediate between the oils of Baku and Pennsylvania as regards the proportion of naphthenes contained (*Ann.*, **220**, 168, 188). According to LE BEL, the oil of Tchongelek, in the Crimea, is largely composed of naphthenes, as are also those of Oelheim and Wietze.

**Naphthenic Acids.**—The earliest investigations of the acid constituents of petroleum were those of HELL and MEDINGER (*Ber.*, **7** (1874), 1216; **10** (1877), 541), who examined the acids obtained from soaps salted out of the alkaline sludges obtained in the refining of the illuminating-oil distillates from Wallachian petroleum. They isolated acids belonging to the series $C_nH_{2n-2}O_2$, and demonstrated that they differed in their properties from the unsaturated acids derived from the series of fatty acids. They more closely resembled the fatty acids, but, on account of their very weak acid character, these authors were led to doubt whether they contained the carboxyl group, and to suggest the possibility of a keto-alcohol or ether-alcohol structure. Later a lacto-alcohol structure was proposed by ZALOZIECKI (*Ber.*, **24** (1891), 1808).

Through the classical researches of MARKOVNIKOV (*Ann. chim. phys.*, (6), **2** (1884), 372; *J. prakt. Chem.*, (2), **49** (1894), 88) and his co-workers it has been shown that the chief constituents of Caucasian petroleum are saturated cyclic hydrocarbons, called naphthenes. A number of acids were isolated from Caucasian petroleum, among which was one identical with an acid of HELL and MEDINGER. MARKOVNIKOV con-

sidered these the monocarboxylic derivatives of the naphthenes. The correctness of this view was demonstrated by ASCHAN ("Chemie der Alicyclischen Verbindungen," **1905,** 508; *Ber.*, **24** (1891), 2710). He converted octonaphthenic acid ($C_8H_{14}O_2$) into its amide, and this into the nitrile ($C_7H_{13}CN$), and also into the amine ($C_7H_{13}NH_2$) He also succeeded in reducing this acid, by the aid of hydriodic acid and phosphorus, to an octonaphthene identical with that found in petroleum.

Attempts were then made both by MARKOVNIKOV and by ASCHAN to synthesize naphthenic acids by the hydrogenation of benzoic acid and the three toluic acids; but the hexahydro-derivatives which they prepared were quite different in their properties from the natural naphthenic acids, and up to recently the cyclohexane carboxylic acids had not been found in petroleum. MARKOVNIKOV (*Ann.*, **307** (1899), 367) has, however, converted the naphthenic acid of the composition $C_6H_{11}COOH$ into the amine $C_6H_{11}NH_2$, which appears to be identical with the synthetically prepared methyl-1-amino-2-cyclo-pentane. It is evident, therefore, that the lower-boiling naphthenic acids are cyclopentane derivatives. Since MARKOVNIKOV considered the acid described by him to be isomeric with the methyl-pentamethylene carboxylic acid (2.1) synthesized by COLMAN and PERKIN (*Ber.*, **21** (1888), 739; see also ZELINSKY, *Ber.*, **25** (1902), 2690), the relation of the side groups to the penthamethylene ring remains in doubt. On isomeric naphthenic acids, see BUSHONG and HUMPHREY, *Orig. Com. 8th Intern. Cong. Appl. Chem.*, **6**, 57.

**Native Coke.**—See *Carbonite.*

**Native Humus Acid.**—See *Humus Acid, Native.*

**Natural Asphalt.**—See *Asphalt.*

**Natural Gas.**—See *Gas, Natural.*

**Natural Paraffin.**—See *Ozokerite.*

**Neft-Gil** (*Neftedegil*).—An abundant material in the naphtha region on Cheleken Island in the Caspian; it is a mixture of paraffins and a resin, and is related to zietrisikite (*J. prakt. Chem.*, **73** (1858), 220 and 321).

**Neudorfite.**—A resin ($C_{18}H_{28}O_2$) which occurs in a coal bed at Neudorf, Moravia (VON SCHRÖCKINGER, *Verh. Geol. Reichs.*, **1878,** 387).

**Neutral Oil.**—(1) An oil of 32° to 36° Bé. gravity, 290° to 318°F. flash-point, and 47 to 81 sec. Saybolt viscosity at 70°F. It is sometimes mixed with animal or vegetable oils.

(2) The oils carrying paraffin which are obtained by the steam-distillation of paraffin-base petroleums after the second-grade illuminating oil has been run off. Neutral oil carrying paraffin is known as "wax distillate."

"Wax distillate" is "cracked," cooled and filter-pressed, for the recovery of paraffin wax, and the oil coming from the presses ("pressed distillate") is reduced to make *viscous neutral oil. Non-viscous neutral oils* are the lighter portions which come off when "pressed distillate" is being reduced to viscous neutral.

The "viscous neutrals" produced from Pennsylvania petroleum possess a gravity range of 30° to 34°Bé. Of the twelve "viscous neutrals" marketed by a prominent refiner, four possessed a gravity of 30.4°Bé., a Saybolt viscosity of 150 at 70°F., a cold test of 15°F., a fire test of 440°F., and a flash-point of 375°F.; four others had a gravity of 30.2°Bé., a Saybolt viscosity of 180 at 70°F., a cold test of 20°F., a fire test of 450°F. and a flash-point of 400°F.; the remaining four, which had a gravity of 30°Bé., possessed a Saybolt viscosity of 200 at 70°F., a cold test of 70°F., a fire test of 475°F. and a flash-point of 410°F.

A "non-viscous neutral" marketed by the same refiner had a gravity of 30°Bé., a Saybolt viscosity of 90 at 70°F., a cold test of 15°F., a fire test of 420°F. and a flash-point of 370°F.

**"Neutralol."**—A proprietary name for liquid petrolatum, *q.v.*

**Newry Pitchstone.**—In Ireland the only occurrences of petroleum appear to be in the pores of igneous rocks that have traversed more or less bituminous strata, and absorbed and modified volatile hydrocarbons in the process. Instances are recorded at Newry (Down) and at the Giant's Causeway. See KNOX, *Phil. Trans.*, **1822**, 313.

**N'hangellite.**—Near Lake N'hangella, in Portuguese East Africa, there is found on the surface of the ground a substance which has been described as elastic bitumen. It is similar to coorongite (*q.v.*) and yields 49.6 per cent. of oil on destructive distillation.

See BOODLE, *Bull. Roy. Bot. Gardens, Kew*, **1907**, 145; and REDWOOD, *ibid.*, 151.

**Nigrite.**—A variety of asphalt mined at Soldier Summit, Utah. Its composition is imperfectly understood.

**Nitronaphthalene.**—*a*-Nitronaphthalene is used to deprive mineral oils of their fluorescence; it is said that 2 to 3 parts are sufficient for 1,000 parts of oil.

On the detection of nitronaphthalene in mineral oils, see LEONARD, *Chem. News*, **68** (1893), 297.

**Nonane.**—Nonane ($C_9H_{20}$) has been isolated from Pennsylvania petroleum (WARREN, *Am. J. Sci.*, (2), **45** (1868), 262).

**Non-Caking Coal.**—A bituminous coal which burns freely without softening or any appearance of incipient fusion. The percentage of volatile matter may be the same as for caking coal (*q.v.*), but the residue is not a true *coke*, being in powder or of the form of the original coal.

**Nonocosane.**—Nonocosane ($C_{29}H_{60}$) was separated by MABERY (*Proc. Am. Acad.*, **40** (1904), 355) during the course of an investigation on the composition of commercial paraffin.

**Nonodecane.**—Nonodecane ($C_{19}H_{40}$) was separated from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565).

**Non-Oleaginous Lubricants.**—In a few exceptional cases oily lubricants are inapplicable. For instance, in chlorine or oxygen compression plants, wherein mineral oils would be chemically attacked, concentrated

sulphuric acid and dilute glycerin respectively are the lubricants used. In the manufacture of sulphur dioxide the liquid dioxide itself acts as a lubricant for the pistons and cylinders.

**Non-Viscous Neutral Oils.**—See *Neutral Oil.*

**"Normal Benzine."**—Benzine of the specific gravity 0.695–0.705 at 15°C. and boiling from 65°–95°C., proposed in Germany for the purpose of detecting and estimating asphalt in petroleum.

**Normal Temperature.**—In laboratory investigations, 25°C. (77°F.).

**"Nujol."**—One of the proprietary names for liquid petrolatum, *q.v.*

**"Obispo."**—This "condensed" or "blown" oil originates in California petroleum; it contains 99.6 per cent. of bitumen soluble in carbon disulphide, 61.5 to 63.4 per cent. of bitumen soluble in 88°Bé. naphtha, 35.7 to 38.2 per cent. of pure bitumen as saturated hydrocarbons, 0.1 to 0.3 per cent. of paraffin scale, 1.22 per cent. of sulphur, and 17.3 to 19.6 per cent. of ash-free residual coke.

**Octane.**—On the octanes in American petroleum, see MABERY and HUDSON, *Proc. Am. Acad.*, **32** (1897), 101.

**Octocosane.**—Octocosane ($C_{28}H_{58}$) was separated by MABERY (*Proc. Am. Acad.*, **37** (1902), 565) during the course of an investigation of the hydrocarbons in Pennsylvania petroleum with boiling points about 216°C. It was also separated by MABERY (*ibid.*, **40** (1904), 355) in an inquiry into the composition of commercial paraffin.

**Octodecane.**—Octodecane ($C_{18}H_{38}$) has been isolated from Pennsylvania petroleum. It was identified by MABERY (*Proc. Am. Acad.*, **37** (1902), 565) among the hydrocarbons in Pennsylvania petroleum with boiling points above 216°C.

**Octonaphthene.**—MARKOVNIKOV (*J. Russ. Phys.-Chem. Soc.*, **16** (1884) 294) isolated octonaphthene from the petroleum of Balakhani and Bibi-Eibat.

**"Oil."**—"Soft paraffin" is commercially known in England as "oil," since it is of no value to the candle-maker.

**Oil Concrete.**—On oil cement concrete, see CUSHMAN, *Science, N. S.*, **31** (1910), 960; and on oil-mixed, Portland cement, mortar and concrete, see PAGE, *Trans. Am. Soc. Civ. Eng.*, **74** (1911), 255.

**"Oildag."**—Deflocculated graphite suspended in oil, used as a lubricating medium (see ACHESON's United States Patent 911358; and ACHESON, *Eng. News*, **53**, 127).

**Oil Fuel.**—Refined or crude petroleum, shale oil, residuum tar, or similar substances, used as fuel.

**Oil-Gas Tars.**—Tars produced by "cracking" oil vapors in the manufacture of oil gas.

**Oil of Paraffin.**—A colorless to yellowish, limpid oil, having a specific gravity of about 0.880 and not boiling below 360°. It is composed principally of high-boiling hydrocarbons of the $C_nH_{2n+2}$ series, and is obtained from the petroleum fraction boiling above 300°, the product

being refined and decolorized. It is used in pharmacy in ointments and as the vehicle for various dermics insoluble in water.

See *Petrolatum* (*Liquid*).

**Oil-Pulp.**—An aluminum soap, consisting of aluminum salts of the fatty acids, chiefly oleic, palmitic and stearic acids. It is dissolved in mineral oil to form an oil thickener.

**Oil-Shale.**—See *Shale*, *Shale Oil*, *Dam-Shale*, *Pumpherston Shales*, and *Broxburn Oil-Shale*, also *Kerogen* and *Bituminous Shales*.

**Okonite.**—For the production of the material of which candles are made, ozokerite is subjected by J. C. & J. Field, Ltd., to a process of distillation, patented in 1870 by Field and Siemssen. Under this process the ozokerite is melted, pumped into stills, and distilled in a current of superheated steam. The distillate is caked, pressed with naphtha, clarified with fullers' earth, and filtered.

The residue in the stills consists of a hard, black, waxy substance, for which at first no use could be found, but in 1875 Field and Talling took out a patent for producing electrical insulating material by combining this black ozokerite with India rubber by passing it through rollers at a moderate temperature, and subsequently vulcanizing the compound. This material, with certain modifications, has been introduced as *okonite* for the insulation of electrical cables. Okonite is not only an insulator, but is flexible and tough. By a similar process, a form of the material known as heel-ball is manufactured. This is employed to impart a black polished surface to the heels and soles of shoes; it is also used by the Austrian Government for the leather work of cavalry and artillery.

**Olefines in Petroleum.**—According to Hofer, the following olefines have been isolated from "North American" petroleum: Ethylene ($C_2H_4$), Propylene ($C_3H_6$), Butylene ($C_4H_8$), Amylene ($C_5H_{10}$), Hexylene ($C_6H_{12}$), Heptylene ($C_7H_{14}$), Octylene ($C_8H_{16}$), Nonylene ($C_9H_{18}$), Decatylene ($C_{10}H_{20}$), Endecatylene ($C_{11}H_{22}$), Dodecatylene ($C_{12}H_{24}$), Decatrilene ($C_{13}H_{26}$), Cetene ($C_{16}H_{32}$), Cerotene ($C_{27}H_{54}$), and Melene ($C_{30}H_{60}$).

On the relative proportions of olefines in shale and petroleum products, see Allen, *Analyst*, **6** (1881), 177.

**Oleo Resin.**—The viscous to semi-solid and usually light colored solutions of true resins in essential oils, either obtained from, or constituting, the saps of certain plants and trees.

**Oleum Asphalti.**—In 1769, T. Healde published a work in London on the "Use of Oleum Asphalti in Ulcers of the Intestines, Lungs, and Viscera."

**"Olo."**—A proprietary name for liquid petrolatum, *q.v.*

**Orchard Heating Oil.**—This product from California petroleum is a dark oil possessing a gravity of 26° to 28°Bé.; it is also termed "smudge oil," and is used in the orange and lemon groves to prevent frost from

injuring the trees. "Smudge oil" from Colorado petroleum usually has a gravity of 30° to 31° Bé.

**Ostatki.**—In the Russian petroleum industry, the residuum left in the still after the distillation of the kerosene is a thin liquid of a specific gravity of about 0.905 to 0.912. It contains but little paraffin, and yields lubricating oils of good quality, but only a small proportion of it (probably about 10 per cent.) is worked up for this purpose, the remainder being utilized for fuel.

The residuum, which is known as ostatki (Russian), commonly misspelt astatki, or masut (Tartar), usually contains from 1 to 2 per cent. of water, although, if less fluid than ordinarily, a larger proportion may be present. It is used as a lubricant for heavy machinery, after separation of water and removal of the unpleasant odor and part of the color by treatment with sulphuric acid; but in order to obtain bright oils of increased viscosity, fractional distillation is necessary, and, as a high temperature is liable to decompose or "crack" the heavy hydrocarbons into lighter and less viscous compounds, superheated steam is employed and occasionally the distillation is conducted *in vacuo*.

**Ozokerite.**—A naturally occurring bituminous product, found in Galicia, Rumania, the Argentine, Orange River Colony, and Utah. It varies in color from yellow to dark brown, and in specific gravity from 0.85–0.97. See BERLINERBLAU'S "Das Erdwachs, Ozokerit, und Ceresin," **1897.** On Utah ozokerite, see *Salt Lake Min. Rev.*, **18,** No. 12, 17; and *Min. Sci. Press*, **112,** 909.

See also *Ceresin*.

**Paint Oil.**—One grade is a sun-bleached neutral oil, pale lemon in color, possessing a gravity of 36°Bé., a flash-point of 260°F., a fire test of 320°F., a cold test of 20°F., and a viscosity of 55. The non-viscous oil described as *concrete form oil* is also used by painters.

**Paint Thinner.**—See *Turpentine Substitutes*.

**Painters' Naphtha.**—See *Turpentine Substitutes*. Deodorized naphtha of gravity 58° to 60° is sometimes employed in paints.

**"Pale Paraffin Oil."**—A petroleum product having a gravity of 25°Bé., a flash-point of 375°F., a cold test of 35°F., and a Saybolt viscosity of 145 to 150 at 100°F.

**Paper Mill Foam Oil.**—Usually identical with *paint oil*. See also *Foam Oil*.

**Paraffin (Native).**—Native crystallized paraffin has been described as occurring in cavities in basaltic lava near Paterno, Sicily (SILVESTRI, *Boll. Com. Geol.*, **12** (1881), 578; *Atti Acc. Gioen. Sci. Nat.*, (3), **12,** 69).

**Paraffin-Asphalt Petroleum.**—A combination of paraffin-base and asphalt-base petroleums. See page 447.

**Paraffin-Base Petroleum.**—Crude oil which carries solid paraffin hydrocarbons and practically no asphalt. See page 447.

**Paraffin Coal.**—See *Earthy Brown Coal*.

**Paraffin Fluxes.**—The residuals obtained from paraffin-base petro-

leums are characterized by containing 14½ to 4 per cent. of hard paraffin scale, consisting to a predominating degree of saturated hydrocarbons (85.6 to 74.1 per cent.) and having a specific gravity of 0.92 to 0.94. Those derived from the paraffin oils of Ohio and Pennsylvania consist of over 80 per cent. of saturated hydrocarbons. In general, it may be said that paraffin fluxes yield only a small percentage of residual coke and contain but little sulphur.

See *Fluxes, Semi-Asphaltic Fluxes, Asphaltic Fluxes and Mexican Fluxes.*

**"Paraffin Oil."**—A name for liquid petrolatum, *q.v.*

Kerosene (*q.v.*) is also so termed.

**Paraffin Oils.**—The lubricating oils made by the dry distillation method are known as paraffin oils. They range in gravity from 30° to 20°Bé., in flash from 300° to 450°F. by the Cleveland open cup, in viscosity from 40 to 600 sec. on the Saybolt viscosimeter at 70°F., in cold test from 0° to 40°F., in color from pale yellow through red, to dark green. Paraffin oils are usually decolorized by sulphuric acid treatment.

In judging the quality of paraffin lubricating oils, the oil that does the work most satisfactorily at the lowest cost is the best product from the consumers' standpoint. The ordinary physical tests should be made, such as gravity, flash and fire tests, cold test, viscosity, and color, and the oil should show no trace of acid. These oils are said to be suitable for all kinds of lubrication, except, perhaps, for steam engine cylinders.

**Paraffin Scale.**—Crude paraffin wax.

**Paraffin Wax.**—The manufacture of paraffin wax is described on pages 478 and 753.

Refined paraffin wax should be practically water-white when in a melted state, colorless, and should not darken rapidly when exposed to the sunlight. It is used to a large extent in the manufacture of candles; it is used also in making so-called wax paper, for waterproofing, for laundering, protecting preserves from fermentation, in admixture with asphaltic products for making insulating pitches for wires, for floors and a variety of other purposes.

See *Paraffinum*, and also pages 476, 483 and 840.

**Paraffinic Acid.**—Paraffinic acid ($C_{24}H_{48}O_2$) is obtained by the oxidation of paraffin by concentrated nitric acid or by chromium trioxide mixture. See POUCHET, *Bull. soc. chim.*, (2), 23, 111.

**Paraffinum.**—According to the "United States Pharmacopœia," paraffinum is described as a mixture of solid hydrocarbons chiefly of the methane series; usually obtained by chilling and pressing the distillates from petroleum having high boiling point, and purifying the solid press-cake so obtained.

Paraffin, in its pure condition, is a white, waxy, inodorous, tasteless substance, harder than tallow, softer than wax, with a specific gravity

of 0.890. Its melting point is variable, depending somewhat upon its origin; it ranges between 43° and 65°C. (109° and 151°F.). An ultimate analysis yields, on the average, carbon 85 per cent. and hydrogen 15 per cent. It is insoluble in water, is indifferent to the most powerful acids, alkalies, and chlorine, and can be distilled unchanged with strong sulphuric acid. Warm alcohol, ether, oil of turpentine, olive oil, benzol, chloroform, and carbon disulphide dissolve it readily. It can be mixed in all proportions with wax, stearin, palmitin, and resin.

**Paraffinum Liquidum.**—The medicinal petroleum of the "British Pharmacopœia." Sp. gr., 0.885–0.89. B.p., above 360°F. In the refining of Russian petroleum, the finest quality of perfumery oil (*q.v.*) is termed "paraffinum liquidum," and for pharmaceutical purposes is often subjected to a final distillation.

See *Petrolatum* (*Liquid*).

**Paraffinum Molle.**—According to the "British Pharmacopœia," a petroleum product corresponding to the vaseline of the "United States Pharmacopœia;" it is described as having a melting point of 95°F.

**Parianite.**—Asphalt (*q.v.*) from the Pitch Lake, Trinidad.

On the nature of parianite, see PECKHAM, *J. Frank. Inst.*, **149** (1900), 161.

**"Parol."**—A fuel for use in internal combustion engines, introduced in1913; it has been reported (*Chem. World*, **2**, 175) that it is "made from paraffin by a chemical process and without the use of heat."

**"Paroline."**—A proprietary name for liquid petrolatum, *q.v.*

**Parrot Coal.**—The original parrot coal was a cannel from near Edinburgh.

See *Cannel Coal.*

**Pavement.**—The wearing course of the roadway or footway, when constructed with a cement or bituminous binder, or composed of blocks or slabs, together with any cushions or "binder" course.

**Peat.**—On the hydrocarbons of peat, see DURIN, *Compt. rend.*, **96** (1883), 652. The manufacture of hydrocarbon oils, paraffin, etc., from peat has been discussed by PAUL, *Chem. News*, **6** (1862), 221 and 243; and by BOHL, *Dingler's polyt. J.*, **140** (1856), 63; *J. prakt. Chem.*, **68** (1856), 504; **75** (1858), 289; *Dingler's polyt. J.*, **144** (1857), 444.

On the fuel value of peat, see B. F. HAANEL, *Rept. No.* **299**, *Mines Branch, Canada Dept. of Mines*, **1914**.

**Peat Tar.**—Peat may be either distilled right down to coke for use in the metallurgical industry or only partially carbonized, so that the residue may find employment as a domestic or industrial fuel. The distillates obtained contain respectively 4 or 2 per cent. of tar, which contains 30 to 40 per cent. of creosote; 40 or 36 per cent. of water-soluble products (ammonia, methyl alcohol and acetic acid); and 21 or 12 per cent. of gaseous products.

**Pelionite.**—A name proposed by W. F. PETTERD ("Catalogue of

Minerals of Tasmania," 1893) for a bituminous coal ("Pelion coal") resembling English cannel coal, from near Monte Pelion, Tasmania.

**Penetration.**—In laboratory investigations, the distance, expressed in tenths of a millimeter, entered a sample by a No. 2 cambric needle operated in a machine for the purpose and under known conditions of loading, time, and temperature. The degree of solidity of bituminous materials.

In construction, the entrance of bituminous material into the interstices of the metal of the roadway.

**Penetration Method.**—The method of constructing a bituminous-macadam pavement by pouring or grouting the bituminous material into the upper course of the road metal before the binding of the latter has been completed.

**"Pennalene White Oil."**—A water-white, odorless oil for medicinal purposes.

**Pentacosane.**—Pentacosane ($C_{25}H_{52}$) has been separated from the hydrocarbons in Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565). It has also been separated from commercial paraffin (MABERY, *ibid.*, **40** (1904), 355).

**Pentadecane.**—Pentadecane ($C_{15}H_{32}$) has been isolated from Pennsylvania petroleum.

**Pentane.**—BEILSTEIN and KURBATOW (*Ber.*, **14** (1881), 1620) found pentane in the lighter distillates from the oil of Zarskije Kolodsy, Tiflis; in Galician oil, LACHOWICZ found normal and isopentane (*Ann.*, **220** (1883), 188); and normal and isopentane have been isolated from Pennsylvania petroleum (see WARREN, *Am. J. Sci.*, (2), **45** (1868), 262). Pentane is now being produced commercially at Whiting, Ind., from Mid-Continent petroleum.

See *Amyl Hydride.*

**Pentatricontane.**—Pentatricontane ($C_{35}H_{72}$) has been separated by MABERY (*Proc. Am. Acad.*, **40** (1904), 349) from the solid hydrocarbons which collect in certain oil wells in Pennsylvania.

**Pentylene.**—See *Amyl Hydride.*

**Perfumery Oil.**—Properly refined Russian perfumery oil possesses a specific gravity of 0.880 to 0.885, is colorless and inodorous, without fluorescence, and does not turn yellow or deposit a sediment after prolonged exposure to direct sunlight. The finest quality is used in pharmacy as "paraffinum liquidum" (*q.v.*).

ROSSMÄSSLER ("Die Petroleum- und Schmierölfabrikation," **1893**, 77) has thus described the processes adopted in Russia in the refining of perfumery and mixing oil:

The distillation product of Russian petroleum-residuum, known as solar oil, is redistilled, the lightest fractions, which are strong-smelling, being rejected. The remainder of the distillate is freed from water by blowing air through it at a temperature of 70°C., until it becomes clear and remains so on cooling. The distillate is then subjected to the acid-

treatment, the first stage of which consists in the addition of 1 to 1½ per cent. of ordinary sulphuric acid. The "sludge acid" having been separated, 10 to 15 per cent. of strong acid—fuming sulphuric acid, or a solution of sulphuric anhydride in ordinary acid, in the proportion of 1 part of 80 per cent. anhydride to 2 parts of acid—is added and agitated with the oil for an hour or an hour and a half, during which time the temperature rises to 35° or 40°C., with evolution of sulphurous acid. The "sludge" is then removed, and the oil is treated with caustic-soda solution (20°Bé.) until faintly alkaline. After removal of the soda sediment, free steam is blown in until the temperature rises to 45° to 50°C., the oil being then left to settle. The aqueous liquid having been drawn off, the oil is repeatedly washed with warm water, air is then blown through it while it is at a temperature of 40°C., and the refining is completed by filtration through bone-black.

**"Petralol."**—A proprietary name for liquid petrolatum, *q.v.*

**"Petro."**—A proprietary name for liquid petrolatum, *q.v.*

**Petrocene.**—The composition of the residues from petroleum-distillation has received considerable attention, more especially as regards Pennsylvania oil. Morton, in 1873 (*Am. Chem.*, **3** (1872), 106 and 162; **7** (1876), 88; and *Chem. News*, **34**, 188), obtained by the distillation of petroleum-residue at a red heat a product which yielded needle-like crystals of a greenish yellow color and pearly luster. This body, which he at first called "viridine," and afterward "thallene," was found to be isomeric with anthracene, though unlike it in crystalline form, melting point, and solubility. Hemilian (*Ber.*, **9** (1876), 1604) asserts that this compound, which he calls "petrocene," has the formula $C_{32}H_{22}$. Tweddle, in 1876, stated that "thallene" is produced by the destructive distillation of a greenish substance, which he termed "petrozcene," obtained from petroleum-residues (*J. Frank. Inst.*, **102**, 204). According to Graebe and Walter (*Ber.*, **14** (1881), 175), "thallene" closely resembles the "picene" obtained by Burg from brown-coal tar. From the least volatile products of petroleum-distillation, Divers and Nakamura (*J. Chem. Soc.*, **47**, 924) have isolated a compound boiling between 280° and 285°C., and possessing the empirical formula $C_4H_3$; while Prunier (*Bull. soc. chim.*, (2), **31** (1879), 293) isolated from the green so-called "petrocene" (boiling point, 190°–240°C.), obtained by distillation of the residues after the ordinary paraffin had come over, hydrocarbons which he called "carbozene," "carbopetrocene," and "thallene." These compounds were found to possess formulas ranging from $(C_4H_2)_n$ to $(C_7H_3)_n$, where $n$ is a variable higher than 4. On "petrocene," see Sadtler and McCarter, *Am. Chem. J.*, **1** (1880), 30.

**Petrol.**—Once used as a synonym for petroleum; now for *petroleum spirit* (see also "*Light Petroleum*" and *Benzine*).

The commercial motor oils, sold in England under the term "petrol," have higher boiling points than petroleum spirit (*q.v.*); as a rule, 60–70

per cent. only boil below 100°C., about 25 per cent. pass over between 100° and 120°C., 5–6 per cent. from 120°–133°C., and about 3 per cent. above 133°C. (see BLOUNT, *J. Soc. Chem. Ind.*, **28** (1909), 419). Petrols containing much larger proportions of higher boiling fractions are, however, now used in modern carburetors.

**Petrolatum.**—Petrolatum, or soft petroleum ointment, is a light yellowish or yellow semi-solid, possessing a specific gravity of 0.820 to 0.850 at 60°C. and melting at 45° to 48°C. It is soluble in ether, chloroform, benzine, carbon disulphide, slightly soluble in alcohol, and insoluble in water and glycerin. Its manufacture is described on page 632.

Petrolatum is widely used in medicine as an antiseptic and emollient; it is a well-known ointment base. Technically, it is employed in clay modelling, as a leather grease, in the lubrication of machinery and firearms, as a shoe polish, in greasing hoofs, as a rust preventive, in the manufacture of soaps and pomades, and in making oiled paper.

**Petrolatum (Liquid).**—A colorless to slightly yellowish, transparent liquid, possessing a specific gravity of 0.840 to 0.940 at 25°C. (usually 0.84–0.87). It is soluble in ether, chloroform, carbon disulphide, benzine, benzol, boiling alcohol, but is scarcely soluble in cold or warm alcohol, and is insoluble in water.

Liquid petrolatum may be made by distilling the residuary liquid boiling between 330° and 390°C., obtained after removing the lighter hydrocarbons from petroleum. It is purified and decolorized by treating it with sulphuric acid and then with caustic soda, and finally passing it, while hot, through bone-black. By cooling, some solid paraffins will separate; the liquid is then redistilled, and the portion boiling below 360°C. is rejected. See also page 634. It has been sold under various proprietary names as "*albolene*," "*glycolin*," etc.; and since its introduction into the "United States Pharmacopœia," 1890, it has been largely used in medicine and for pharmaceutical purposes.

Liquid petrolatum is used in medicine as an emollient; it is a well-known vehicle for antiseptic remedies, and is also used in the treatment of constipation.

Most of the so-called "paraffin oils" contain no paraffin, being made up of the naphthene and poly-naphthene classes.

On liquid petrolatum, see *J. Am. Med. Assn.*, Mar. 6, **1915**, 808; May 29, **1915**, 1853; and July 10, **1915**, 175; *J. Pharm. Chim.*, **9** (1914), 149; GANE, *J. Am. Pharm. Assn.*, **4** (1915), 792; HILTON, *ibid.*, 990; and REMINGTON, *ibid.*, 933.

See *Petrolatum Oil* and *Spindle Oil*. Cf. *Perfumery Oil*.

**Petrolatum (Veterinary).**—Veterinary petrolatum is a dark yellow, semi-solid mass; it is essentially crude (unpurified) petrolatum.

This petrolatum is employed in veterinary surgery as an ointment base.

**Petrolatum (White).**—This preparation, also known as "white vaseline" and "albolene," which are proprietary brands, is manufactured from yellow petrolatum by depriving the latter of its color by bleaching. It is a white semi-solid, possessing an ointment-like consistency, and is easily soluble in ether, chloroform, benzine, and carbon disulphide; but is only slightly soluble in alcohol, and is insoluble in water and in glycerin. It possesses a melting point of 40° to 45°C.

White petrolatum is used as an ointment base, in extracting perfumes, in modelling clay, softening leather, greasing firearms, lubricating machinery, as a rust preventive, and in the manufacture of soaps, shoe polishes and putz pomades.

**Petrolatum Liquidum.**—The medicinal high-boiling petroleum oil of the "United States Pharmacopœia." See *Petrolatum (Liquid)*.

**Petrolatum Oil.**—A white, straight-reduced viscous neutral oil, possessing a gravity of 32¾° to 34°Bé., a flash-point of 415°F., a fire test of 480°F., a cold test of 20°F., and a viscosity of 185 to 200. It is also termed "medicinal oil."

See *Petrolatum (Liquid)*.

**Petrolatum Spissum.**—See *Petrolatum*.

**"Petrolax."**—A proprietary name for liquid petrolatum, *q.v.*

**Petrolene.**—Boussingault (*Ann. chim. phys.*, **64** (1837), 141; **73** (1840), 442) obtained from the viscid bitumen and asphalt of Pechelbronn an oil which he termed "petrolene;" he announced it as the liquid ingredient of all asphalt, the solid one being termed by him asphaltene (*q.v.*). "Petrolene" was separated by heating asphalt to 300°C.; it boiled at 280°C.

**Petroleum.**—Petroleum, the best known of the bitumens, and, next to coal, the most important of all carbon compounds, is an oily, inflammable liquid mixture of hydrocarbons occurring naturally, oozing from crevices in rocks, floating on the surface of water, or in subterranean deposits in rocks, from which it may be obtained by boring. See page 1.

Petroleum is sometimes used in the crude state for fuel, for surfacing roads, or for the prevention of dust on roads; but generally the first stage in the utilization of crude petroleum for any purpose is to subject it to some form of distillation.

In general, there are three kinds of petroleum, namely: paraffin-base, which carries solid paraffin hydrocarbons and practically no asphalt; asphalt-base, which contains asphalt and no paraffin; and paraffin-asphalt, a combination of the two. The paraffin-base petroleum is usually of lightest gravity and it yields the largest variety of lubricating oils.

**Petroleum Acids.**—See *Naphthenic Acids*.

**Petroleum Asphalt.**—The residues of asphalt-base petroleums are known commercially as petroleum asphalt. These may be steam residues, dry or oxygenated residues. Asphaltoid products are also made

from the sludge acids resulting from the treatment of paraffin oils with sulphuric acid, or they may be made by air-blowing distillates at high temperatures. Petroleum asphalts are used in street paving, waterproofing, and in the manufacture of roofing pitches.

See *Asphalt*, *"Condensed" or "Blown" Oils*, and *Pitch*.

**Petroleum Benzin.**—The "petroleum benzin" of the "German Pharmacopœia" consists of the colorless, nonfluorescent portions of petroleum, possessing a specific gravity of 0.640 to 0.670, and distilling almost entirely between 55° and 75°C.

The petroleum naphtha or benzine of Russian refiners varies in boiling point from 80° to 100°C. and in specific gravity from 0.667 to 0.707 at 15°C.

See *Benzine*, *Light Petroleum* and *Canadol*.

**Petroleum Briquet.**—One type of briquet is made of a mixture of 1 liter of petroleum, 150 grams of soft soap, 150 grams of resin, and 300 grams of "soda-lye wash." This mixture is well heated, allowed to solidify, run into moulds, and then heated in a furnace for about 15 min. The solidity can be increased by adding sawdust or sand.

**Petroleum-Butter.**—On petroleum-butter, see NEUBURGER, *J. Pétrole*, 9 (1909), 209.

**Petroleum-Coke.**—See *Coke*.

**Petroleum Distillate.**—See *Power Distillate*.

**Petroleum Ether.**—Some refiners have applied this designation to the products ranging in specific gravity from 0.590 to 0.666 (108° to 80°Bé.), that is, *cymogene*, *rhigolene*, and *gasoline*. See *Benzine*, *Canadol*, *Light Petroleum*, *Keroselene*, and *Sherwood Oil*. Russian petroleum ether varies in specific gravity from 0.650 to 0.660 at 15°C.

**Petroleum Jelly.**—See *Cosmoline*, *Petrolatum*, and *Vaseline*.

**Petroleum Naphtha.**—A term which is loosely employed; it often denotes the first fraction (b.p., up to 150°C.) obtained on distillation of crude oil, but is also applied to any low-boiling petroleum product.

See *Naphtha* and *Benzine*, also *Petroleum Benzine*.

**Petroleum Ointment.**—See *Petrolatum*.

**Petroleum Pitch.**—See *Pitch*.

**Petroleum Spirit.**—A term which is variously used, but is sometimes applied to a petroleum distillate of a density of 0.71 to 0.74 and a boiling point of 90° to 140°C. It is used as a solvent (see *gasoline*).

Some refiners have employed the term to include naphthas ranging in specific gravity from 0.679 to 0.745 (76° to 58°Bé.).

According to German practice, petroleum spirit used as motor fuel should contain nothing, or at the very most 5 per cent., boiling above 100°C. Cf. *Petrol*.

See *Benzine*, *Benzoline*, and *Naphtha*.

**Petroleum Tailings.**—See *Residuum*.

**"Petrolia."**—A proprietary name for liquid petrolatum, *q.v.*

**Petroline.**—A solid substance, analogous to paraffin, obtained in the distillation of Rangoon petroleum.

Also, a term applied to a Scottish burning oil having a flash-point of 126°F.

**"Petronol."**—A proprietary name for liquid petrolatum, *q.v.*

**"Petrosapol."**—This is a brown ointment-like compound of soap and the by-products of petroleum; it is especially used as a vehicle for ointments. It does not melt below 90°F. and does not therefore liquefy on the skin.

**"Petrosio."**—A proprietary name for liquid petrolatum, *q.v.*

**Petrozcene.**—On petrozcene and its products, see TWEDDLE, *J. Frank. Inst.*, **102** (1876), 204. See also *Petrocene.*

**"Pharmaceutic Naphthene Oil."**—A designation of *liquid petrolatum.*

**Phenol in Petroleum.**—PEBAL (*Ann.*, **115** (1860), 19) found phenol in Galician petroleum. MARKOVNIKOV was the first to find phenol in crude Caucasian petroleum, and, in conjunction with OGLOBLIN, investigated the composition of the oxygen acids of petroleum. MARKOVNIKOV found as much as 5.25 per cent. of oxygen in the fraction of Caucasian petroleum boiling between 220° and 230°C. He considers the acids to be carboxylic acids, derived from the naphthenes of the petroleum; but ZALOŻIECKI (*Z. angew. Chem.*, **4** (1891), 416) states that they consist mainly of *acid lacto-alcohols.* ASCHAN (*Ber.*, **23** (1890), 867; **24** (1891), 2710) confirms MARKOVNIKOV's contention that they are naphthene derivatives, and has prepared octonaphthene from one of them. According to KRÄMER, however, these acids are ordinary *fatty acids.* C. J. ROBINSON (*J. Soc. Chem. Ind.*, **1899**, 232) more recently gives great support to this statement by the discovery of acetaldehyde in both Pennsylvania and Ohio petroleums. He found as much as 0.001 per cent. of acetaldehyde in the crude oil. ENGLER (*Verh. Ver. Beförd. Gewerbfl. Preuss.*, **66** (1887), 637) has detected phenols and fatty acids in several German oils.

See *Naphthenic Acid.*

**"Phinotas Oil."**—An insecticide containing coal-tar phenols and light petroleum distillate (*U. S. Dept. Agr., Div. Entom., Bull.* **46**, 108).

**"Photogen."**—A name for *kerosene.*

**"Photogene."**—A trade-name for a shale-oil distillate, of specific gravity from 0.72–0.81 and boiling point from 145°–150°C. It is used as an illuminating oil.

**Phylloretin.**—A resin obtained from an alcoholic solution of a resin from the marshes near Holtegaard in Denmark (FORCHHAMMER, *J. prakt. Chem.*, **20** (1840), 459).

**Phytocollite.**—A black gelatinous hydrocarbon, related to *dopplerite* (COOPER, *Eng. Min. J.*, **32** (1881), 103), found in a stratum of muck below a peat bed at Scranton, Pa.

**Phytosterin.**—On the separation of phytosterin from mineral oils,

see MARCUSSON, *Mitt. k. techn. Versuchsanst.*, 18 (1900), 261; 19 (1901), 259.

**Piauzite** (*Piausit*).—An asphaltoid substance, melting at 315°C.; it has a brownish or greenish black color and a specific gravity of 1.220. After fusing, it burns with an aromatic odor and leaves about 6 per cent. of ash. It is soluble in potassium hydroxide and in ether. It was obtained by HAIDINGER (*Ann. phys. chem.*, 138 (1844), 275) from a bed of brown coal at Piause, near Neustädt, in Carniola.

**Picolene.**—On the presence of *a*-picolene in brown coal tar, see FRESE, *Z. angew. Chem.*, 16 (1903), 11.

**Pissasphalt.**—See *Maltha*.

**Pit Car Oil.**—This is usually of the grade described as *summer black oil*.

**Pitch.**—Solid residue produced in the evaporation or distillation of bitumens, the term being usually applied to residue obtained from tar.

Pitch may be more fully described as a dark colored, bituminous substance, liquid or semi-liquid at room temperature, often possessing a characteristic "tarry" odor, usually insoluble in water, but miscible with carbon disulphide, benzol, etc., and which on distillation, oxidation, etc., forms a pitch. Its composition and origin are variable.

(*a*) *Natural.*—Resulting from a slow natural process of metamorphosis, known as "mineral pitches" or natural asphalts; Trinidad pitch, glance pitch, gilsonite, etc.

(*b*) *Artificial.*—1. Residues from the distillation, oxidation, etc., of mineral oils; *e.g.*, petroleum pitch, blown petroleum asphalt, sludge pitch, Dubbs' asphalt, etc.

2. Residues from the distillation of tars; *e.g.*, coal-tar pitch, brown-coal-tar pitch, coke-oven-tar pitch, blast-furnace-tar pitch, water-gas-tar pitch, generator-gas-tar pitch, wood-tar pitch, pine-tar pitch, etc.

3. Residues from the distillation of fusible organic substances, the process having been terminated before the actual formation of coke; *e.g.*, resin pitch, stearin pitch (fatty acid pitches), etc.

4. Artificial mixtures complying with the above general definition of the term "pitch," regardless of their origin or composition (*e.g.*, insulating pitch, brewer's pitch, roofing pitch, etc.).

See *Residual Pitches*.

**Pitch Coal.**—This lignite, also known as specular coal, is compact, more or less brittle, and occasionally tough, but seldom hard. Dark brown to pitch black, with a waxy or somewhat pitchy gloss. Fracture, imperfect; sometimes flat, conchoidal. Powder, brown. Hardness, 2.5. Specific gravity, 1.2 to 1.3. Pitch coal occurs sometimes alone, sometimes as streaks in the seams of common brown coal or the earthy brown coal beds.

**"Pittsburgh Flux."**—This "condensed" or "blown" oil originates in Ohio petroleum; it contains 97.6 per cent. of bitumen soluble in carbon

disulphide, 66.1 per cent. of bitumen soluble in 88°Bé. naphtha, 55.5 per cent. of pure bitumen as saturated hydrocarbons, 3.7 per cent. of paraffin scale, 4.75 per cent. of sulphur, and 13.7 per cent. of ash-free residual coke.

**Plumbers' Furnace Gasoline.**—A gasoline of gravity 66° to 68°Bé.

**Pneumatic Oil.**—Oil for pneumatic tools is generally of two grades:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 32° | 400°F. | 460°F. | 20°F. | 145 |
| 34° | 340° | 400° | 20° | 80 |

**Pocket.**—A hole or depression in the wearing course of a pavement.

**"Polarine."**—A lubricating oil for motors. See *Motor Oil.*

**Polishing Oil.**—A term applied to that fraction of b.p. 130°–160°C., obtained in refining petroleum.

Also a non-viscous neutral oil identical with *concrete form oil.*

**Posepnyte.**—An oxygenated hydrocarbon from the Great Western mercury mine, Lake Co., Cal. It occurs in plates and nodules, sometimes brittle, occasionally hard; the color is light green to reddish brown; and the specific gravity ranges from 0.85 to 0.985. See BECKER, *Mon. U. S. Geol. Survey*, **13**, 361 (1888).

**Pot-Hole.**—A hole extending below the wearing course in a pavement.

**Power Distillate.**—The untreated kerosene condensates and still heavier distillates down to 28°Bé. from Mid-Continent petroleum, used as fuel in internal combustion engines.

**"Pressed Distillate."**—The oil coming from the presses when paraffin wax is recovered. See *Neutral Oil.*

**"Prima Oil."**—The trade-name of the grade of "solar oil" (*q.v.*) from shale oil with a low density and boiling point.

**"Prime City Naphtha."**—A petroleum product which possessed a gravity of from 73° to 68°Bé.; it was used for sponge-lamps. See *Benzoline.*

**Prime White Oil.**—A kerosene of prime white color, that is, intermediate in color between water-white and standard white. See *Standard White Oil.*

**Propane.**—Propane ($C_3H_8$) is one of the gaseous paraffins which have been isolated from Pennsylvania petroleum.

**Psathyrit.**—See *Xyloretinite.*

**Pseudocumene.**—Pseudocumene has been found in Caucasian oil by MARKOVNIKOV and OGLOBLIN (*J. Russ. Phys.-Chem. Soc.*, **13**, 179; **14**, 36; **15**, 237 and 307) and in the petroleum of America, Germany, Galicia, and Italy by ENGLER (*Ber.*, **18** (1885), 2234).

See *Cumene* and *Mesitylene.*

**Pumpherston Shales.**—These Scottish oil shales yield, on the average, 16 to 22 gal. of crude oil per ton, together with 50 to 60 lb. of ammonium sulphate. Their particular feature is their richness in nitrogen. See page 813.

**Putty Oil.**—The products described under both *concrete form oil* and *paint oil* are sold also as putty oil.

**"Putz" Oil.**—A light distillate of Baku petroleum, possessing a specific gravity of 0.750 to 0.770; it is used as a cleansing oil.

**Pyrène.**—A hydrocarbon ($C_{16}H_{10}$), obtained from coal tar. It was described by LAURENT in *Compt. rend.*, **5** (1837), 803.

**Pyridine in Shale Naphtha.**—On the presence of pyridine in naphtha, see WILLIAMS, *Phil. Mag.*, (4), **8** (1854), 209.

**Pyrimont Bitumen.**—On this bitumen, see PUVIS, *Ann. Mines*, (3), **6** (1834), 179; and ROZET, *Bull. Soc. Géol. France*, **7** (1835), 136.

**Pyrobitumen.**—A dark colored, solid, infusible, naturally occurring hydrocarbon complex, often associated with a mineral matrix, insoluble in water, and relatively insoluble in carbon disulphide, benzol, etc.

Pyrobitumens are derived from the metamorphosis of vegetable growth (*e.g.*, peat, lignite, bituminous coal and anthracite coal), and from the metamorphosis of asphalts (*e.g.*, elaterite or wurtzilite, albertite and impsonite).

**Pyronaphtha.**—This fraction of Russian petroleum is also known as heavy illuminating oil and solar oil; it possesses a light yellow color and a specific gravity of 0.840 to 0.860. However, the product to which the name of pyronaphtha is given, is usually of a lower gravity than solar oil (*q.v.*).

**Pyropissite.**—An earthy, friable, coaly substance, of a grayish brown color and having a specific gravity of 0.493–0.522; it melts easily to a pitch-like mass and affords 62 per cent. of paraffin on dry distillation. Pyropissite occurs in brown coal at Weissenfels, near Halle, Germany (KENNGOTT'S "Uebersichte," **1850**, 148).

**Pyroretin.**—A resin which occurs in brown-coal, between Saleel and Proboscht, near Aussig in Bohemia; it is brittle, of a brownish black color, and has a specific gravity of 1.05 to 1.18 (REUSS, *Ber. Akad. Wien*, **12** (1854), 551).

**Pyroretinite.**—A resin-like substance, deposited from a hot alcoholic solution of *pyroretin* as it cools (STANEK, *Ber. Akad. Wien*, **12** (1854), 554).

**Pyroscheererite.**—A distillation product of könlite (*q.v.*); it is mentioned by KRAUS in *Ann. phys. chem.*, **43** (1838), 141.

**Raeburn Shale.**—See *Dam Shale.*

**Rangoon Oil.**—The trade-name of a semi-solid or butter-like petroleum from Upper Burma, refined at one time in England.

For the results of chemical examinations of the petroleum of Rangoon, see CHRISTISON, *Trans. Roy. Soc. Edinb.*, **13** (1836), 118; GREG-

ORY, *ibid.*, 124; and WARREN and STORER, *Mem. Am. Acad.*, (2), **9** (1867), 208.

**Recovered Oils.**—For reasons of economy, used lubricating oils are collected and used again after purification, usually by filtration. Such "recovered oils" are often darker in color than the original oils, but, when the purification has been carried out in an efficient filter, are said to be equally satisfactory.

**Reduced Fuel Oil.**—Fuel oil of heavy gravity. One having a gravity of 23°Bé. (7.62 lb. per gallon) has been found to contain 19,800 B.t.u. per pound.

**Reduced Oil.**—Crude petroleum from which the more volatile hydrocarbons have been eliminated by partial evaporation. See page 527.

**Refikite.**—A white, very soft resin ($C_{20}H_{16}O_2$) found in the lignite of Montorio, near Feramo, Abruzzes, Italy (DESCLOIZEAUX's "Manuel de Mineralogie," 1874, **2**, 58).

**Refined Tar.**—A tar freed from water by evaporation or distillation which is continued until the residue is of desired consistency, or a product produced by fluxing tar residuum with a tar distillate.

See *Pitch* and also *Flux*.

**Repress Oil.**—Brick repress oil is compounded of 80 per cent. of the neutral oil described under *paint oil* and 20 per cent. of castor machine oil (*q.v.*).

**Residual Pitches.**—The solid residual pitches which are used for highway construction, first originated in California petroleum, but were soon followed in the eastern market by others derived from Beaumont, Texas, oil and later petroleum from other Texas fields. The Texas residual pitches are characterized by containing a small percentage of paraffin scale, less than 1.25 per cent. of sulphur, and yielding, on ignition, a very high percentage of ash-free residual coke; the amount of saturated hydrocarbons which they contain is very much smaller than that of the fluxes, which are intermediate products in their preparation. The California residual pitches are distinguished from those derived from Texas petroleum to a marked degree, in that the percentage of saturated hydrocarbons present is much smaller than in the case of the Texas residual pitches and the amount of ash-free residual coke is also smaller; they are devoid of more than a trace of paraffin scale and run less than 1.5 per cent. of sulphur. Trinidad residual pitches are differentiated from the California products by the higher percentage of sulphur (about 2 per cent.) and by the smaller percentage of saturated hydrocarbons which they contain, lower in amount than is the case in the California residual pitches and approaching in the latter respect the solid native asphalt. Mexican residual pitches are strikingly differentiated from those produced from other petroleum, in that they contain from 5 to 6 per cent. of sulphur, yield in the more solid forms a high percentage of ash-free residual coke, and at the same time contain from

1 to 2 per cent. or more of hard paraffin scale; these pitches, which are unique as compared to the others, are also characterized by the difficulty encountered in their preparation.

See *Pitch*, and RICHARDSON'S "The Modern Asphalt Pavement," 2nd ed., 256–77.

**Residuum.**—1. The residue obtained on the distillation of crude petroleum after the constituents boiling below 620°F. have been removed. The yield of residuum from Pennsylvania petroleum amounts to 12–14 per cent.

2. The residue left in the still after the distillation of crude oil has been completed, and not the residue from redistilled condensates. See page 461.

The residuum from petroleum distillation is also known as the "cokings."

See *Pitch.*

**Resin from Naphtha and Bitumens.**—On the treatment of naphtha and bitumens for the purpose of obtaining solid resin and aromatic hydrocarbons, see ADIYASIEVITCH, *Zap. Imp. Russ. Tekhn. Obsch.*, **29** (1895), 93.

Cf. "*Condensed*" or "*Blown*" *Oils.*

**"Resiniferous Shale."**—See *Tasmanite.*

**Resin, Mineral.**—A term applied to the solid bitumens. See *Bitumen.*

**Resin, True.**—A term applied to various vegetable principles occurring in, or obtained from, the secretions or saps of certain plants or trees. They are hard, fusible and more or less brittle, nonadherent to slightly adherent solids at ordinary temperatures, usually light colored in mass, having an amorphous structure, conchoidal fracture and "resinous" luster. They are insoluble in water, but more or less completely soluble in carbon disulphide, benzol, etc. They are oxidation or polymerization products of the terpenes, and generally contain "resin" acids and esters.

Cf. *Bitumen.*

**Resinous Substance.**—A term applied to (*a*) substances containing true resins; or to (*b*) substances of variable origin and composition resembling true resins in their *physical* properties (with the exception of color) and solubility.

**Retene.**—A crystalline hydrocarbon ($C_{18}H_{18}$), found in certain resins.

On retene, see PECKHAM, *J. Frank. Inst.*, **151** (1901), 50; and SCHMIDT, *Arch. Pharm.*, (3), **6** (1875), 538.

**Retinasphaltum.**—The "retinasphalt" of HATCHETT (*Phil. Trans.*, **94** (1804), 402) was found in the Tertiary coal of Bovey in Devonshire, England; it had the specific gravity 1.135, varied in color from light yellowish brown to red, and yielded a resin (see *retinellite*) on extraction with alcohol.

On this bitumen, see FINCH, *Quart. J. Sci. Lett. Arts*, **11** (1821), 221;

JOHNSTON, *Phil. Mag.*, (2), **12** (1838), 389; and MICKSCH, *Corr.-Bl. zool.-min. Ver. Regensb.*, **1847**, 70.

**Retinellite.**—A light brown, resinoid substance separated by alcohol from *retinasphalt;* it begins to fuse at 121°C. This is the "retinic acid" of JOHNSTON (*Phil. Mag.*, (2), **12** (1838), 560).

**Retinic Acid.**—See *Retinellite.*

**Retinite.**—A general name applied to various resins, especially those from beds of brown coal, which are near succinite in appearance, but contain little or no succinic acid (BUCHOLZ, *J. chem. phys.*, **1** (1811), 290; DIETRICH, *Verh. Geol. Reichs.*, **1875**, No. 8; ZENGELIS, *Min. petr. Mitth.*, **20** (1901), 356). See *Walchowite, Gedanite, Glessite, Rumanite,* and *Simetite.*

**Reussinite.**—A resin-like, reddish brown oxygenated hydrocarbon, soluble in boiling alcohol and in ether. STANEK (*Ber. Akad. Wiss. Wien*, **12**, 554) regarded it as a mixture of pyroretinite (*q.v.*) with another resin, $C_{40}H_{56}O_2$.

**"Rezilite Mastic."**—A preparation of elaterite (*q.v.*), containing some asbestos fibre; it is used for surfacing floors.

**Rhigolene.**—The most volatile liquid fraction obtained in the distillation of petroleum. It has a boiling point of 18°C. and consists largely of pentane (KHARITCHKOV, *Nepht. Dielo*, **1899**, 170); its specific gravity is 0.60.

This petroleum ether was introduced for producing local anesthesia by freezing (BIGELOW, *Chem. News*, **13** (1866), 244). In "æther anæstheticus König" four parts of rhigolene are mixed with one part of absolute ethyl ether.

The term rhigolene has also been applied to the product described under *cymogene;* specific gravity, 0.625 to 0.636. Crude naphtha from Pennsylvania petroleum yields about 0.1 per cent. of such rhigolene.

**Road Binders.**—This group of products consists of petroleum asphalt, properly fluxed with heavy petroleum oils which will not evaporate and which are of such qualities that they will bind the road materials together both in summer and winter.

**Road Oil.**—Road oil from Pennsylvania petroleum usually possesses a gravity of 34° to 35°Bé., a viscosity of 40 at 70°F. by the Saybolt instrument, a fire test of 340°F., and a flash-point of 300°F.

For a full account of the grades of oil covered by this trade-name in the Mid-Continent field, see page 493.

See *Flux Oil, Asphaltic Fluxes,* and *Petroleum.* Cf. *"Kiton," Water-Soluble Oils,* and *Westrumite.*

**Rochlederite.**—A resinoid, reddish brown oxygenated hydrocarbon, found in brown coal in Bohemia; it melts at 100°C. and is soluble in alcohol (ROCHLEDER, *Ber. Akad. Wiss. Wien*, **6** (1851), 53; HAIDINGER, *Lotos*, **1**, 85, 216; **6**, 86; **8**, 3). See *Melanchym* and *Melanellite.*

**Rock Asphalt.**—Sandstone or limestone naturally impregnated with asphalt.

**Rock Asphalt Pavement.**—A wearing course composed of broken or pulverized rock asphalt, with or without the addition of other bituminous materials.

**"Rock Oil."**—A name for liquid petrolatum, *q.v.*

Also a synonym for petroleum.

**"Rod Wax."**—A light-yellow, pasty mass consisting of an emulsion of high-boiling oils with solid hydrocarbons; it collects in considerable quantities around the rods and casing in some of the Pennsylvania wells.

**Roll Oil.**—Oil for tin, copper and brass rolls is usually of the same grade as *engine oil.*

**Rosin Oil Adulterant.**—A viscous neutral oil of the gravity 32°Bé. and the viscosity 145, and a non-viscous neutral oil of a grade identical with *concrete form oil,* have both been much used to replace rosin oil.

**Rosin Oils.**—Heavy rosin oils, boiling above 300°C., are produced by the destructive distillation of colophony, light rosin spirit or pinolin coming over in the first runnings; they may contain, when crude, up to 30 per cent. of rosin acids. Crude and refined rosin oils find application as electrical insulators, and in the manufacture of lubricants, varnishes, and water-soluble oils (*q.v.*).

**Rosthornite.**—A resin ($C_{24}H_{40}O$) possessing a brown color and a specific gravity of 1.076, found at Sonnberge, Carinthia (HÖFER, *Jahrb. Min.*, **1871**, 561).

**Rubber from Petroleum.**—See page 805.

**Rubbing Oil.**—A non-viscous neutral oil of the same grade as *concrete form oil.*

**Rumanite.**—A yellow amber-like resin obtained from different points in Rumania (HELM, *Schrift. Ges. Danzig,* **7** (1891), 186).

On Moldavian rumanite, see ISTRATI, *Bull. Soc. Sci. Bucharest,* **4** (1895), 59; **6** (1897), 55; **7** (1898), 272; **9** (1901), 650.

**"Russian Liquid Petrolatum."**—A name for liquid petrolatum, *q.v.*

**"Russian Mineral Oil."**—A name for liquid petrolatum, *q.v.*

**"Russian Paraffin Oil."**—A name for liquid petrolatum, *q.v.*

**"Russol."**—A name for liquid petrolatum, *q.v.*

**Saponated Petroleum.**—Liquid saponated petroleum may be prepared from 100 parts of liquid petrolatum, 50 parts of oleic acid, and 25 parts of spirits of ammonia. Solid saponated petroleum is compounded of 100 parts of petrolatum, 50 parts of oleic acid, and 25 parts of spirits of ammonia.

**"Satin Gloss Black."**—A name for *lampblack.*

**Saturating Oil.**—This product is identical with the oil described under *concrete form oil.*

**"Saxol."**—A proprietary name for liquid petrolatum, *q.v.*

**"Saxoline."**—A proprietary brand of petrolatum, *q.v.*

**"Scale."**—Scotch crude paraffin wax is known in commerce under this name.

**Scheererite.**—A whitish, gray, yellow, green or pale reddish, easily frangible, tasteless, inodorous hydrocarbon; it melts at 44°C. and is soluble in alcohol and ether. It may be distilled without decomposition, boiling at 92°C.

On the scheererite of Utznach, see KRAUS, *Ann. phys. chem.*, **43** (1838), 141; on the scheererite of Westerwald, see NOEGGERATH, *J. chem. phys.*, **63** (1831), 459; and on scheererite in general, see STROMEYER, *Arch. ges. Nat.*, **10** (1827), 113.

**Schlanite.**—See account of *anthracoxenite*.

**Schraufite.**—A resin ($C_{11}H_{16}O_2$) which occurs in Carpathian sandstone near Wamma, in Bukowina; it has a specific gravity of 1.0 to 1.12 and fuses at 326°C. (VON SCHRÖCKINGER, *Verh. Geol. Reichs.*, **1875**, 134).

**Scleretinite.**—A black, brilliant oxygenated hydrocarbon from the coal measures of Wigan, England; it has a specific gravity of 1.136, and is insoluble in alcohol, ether, alkalies and dilute acids (MALLET, *Phil. Mag.*, **4** (1852), 261).

**Screw-Cutting Oil.**—Oil adapted for heavy lathe and thread-cutting purposes is generally compounded with cottonseed oil or pure lard oil. Screw-cutting oils are often mixtures of 27°Bé. paraffin and 25 per cent. cottonseed oil.

**Seal Coat.**—A final superficial application of bituminous material during construction to a bituminous pavement.

**"Secunda Oil."**—A trade-name of the grade of "solar oil" from shale oil with a high density and boiling point.

**Semi-Asphaltic Fluxes.**—The semi-asphaltic fluxes are differentiated primarily by a higher density than those derived from paraffin-base petroleums, namely, 0.95 to 0.97. They contain a much smaller amount of paraffin scale and approach very nearly in the amount of saturated hydrocarbons which are present to the paraffin residuals. They will satisfactorily dissolve and flux such solid bitumens as gilsonite and grahamite.

See *Fluxes, Paraffin Fluxes, Asphaltic Fluxes* and *Mexican Fluxes*.

**Semi-Solid Bituminous Material.**—Bituminous material showing a penetration at normal temperature under a load of 100 grams applied for 5 sec. of more than 10, and under a load of 50 grams applied for 1 sec. of not more than 350.

**"Semprolin."**—A proprietary name for liquid petrolatum, *q.v.*

**"Seneca Oil."**—An early name for *petroleum*.

**Separator Oil.**—See *Cream Separator Oils*.

**Setting Up.**—The relatively quick change such as takes place in a bituminous material after its application to a roadway, indicated by its hardening after cooling and exposure to atmospheric and traffic condi-

tions, as opposed to the slower changes later occurring gradually and almost imperceptibly.

**Settlingite.**—See *Settling Stones Resin.*

**Settling Stones Resin.**—A resinoid, hard, brittle substance, possessing a pale yellow to deep red color and a specific gravity of 1.16 to 1.54, and burning in a candle flame. It was found in an old lead mine in Northumberland, England (JOHNSTON, *Phil. Mag.*, (2), **14** (1839), 88).

**Sewing Machine Oil.**—The following neutral oils are recommended for sewing machine lubrication:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 34°Bé. | 340°F. | 400°F. | 20°F. | 80 |
| 34½° | 340° | 400° | 20° | 75 |
| 35° | 340° | 400° | 20° | 70 |

**Seyssel-Pyrimont Bitumen.**—Ascending the right bank of the Rhone, the Urgonian limestone recurs, with the famous "Seyssel" asphalt of Pyrimont, extending by Perte du Rhone and Bellegarde, to Thoiry, and up the Valserine valley from Bellegarde by Lancrau to Lelex and Forens, near Chézery. Seyssel asphalt rock consists of 89.55 per cent. of calcium carbonate and contains 8 per cent. of a portion soluble in carbon disulphide.

This bitumen is described by MASSAT, *Le Naturaliste*, **19** (1897), 17; and URE, *London J. Arts*, (3), **14** (1839), 251.

**Shafting Oil.**—See *machine oil*, with which shafting oil is identical. *Summer black oil* is also used for shafting lubrication.

**"Shale," or Oil-Shale.**—A highly bituminous laminated substance containing about 80 per cent. of mineral matter, occurring in many parts of the world, but especially in the coal measures of the Lothians in Scotland. See page 807.

See also *Bituminous Shales.*

**Shale Naphtha.**—The naphtha from shale oil generally consists of 60 to 70 per cent. of olefines and other hydrocarbons acted on by fuming nitric acid. It is obtained at different specific gravities and boiling points according to requirements; for instance, 0.660 for gasoline, 0.690 for motor spirit, and 0.72 to 0.75 for lighting purposes and as solvents. See page 840.

Cf. *Shale Spirit.*

**Shale Oil.**—A crude oil obtained from bituminous shales, especially in Scotland, by submitting them to destructive distillation in special retorts. It is of a dark green to a brownish color, possesses a specific gravity of 0.86 to 0.96, and has a congealing point of about 32°F. Its quality depends greatly on the temperature at which it is formed from

the kerogen, or bituminous matter, in the shale (the greater part of the decomposition and distillation in the retorts occurs below 427°C.). Shale oil is refined into products resembling those from petroleum. On the technology of the Scottish shale oil industry, see page 814.

**Shale Spirit.**—The lower-boiling fractions obtained in the refining of crude shale oil; sp. gr., 0.70–0.76. It is used as a motor spirit and contains about 50–60 per cent. of unsaturated hydrocarbons. See page 840.

**Sheet-Asphalt Pavement.**—One having a wearing course composed of asphalt cement and sand of predetermined grading, with or without the addition of fine material, incorporated together by mixing methods.

**Sherwood Oil.**—Petroleum ether boiling at 40° to 70°C.

See *Light Petroleum.*

**"Sicherheitsbenzin."**—A mixture of 1 volume of benzine and 2 volumes of carbon tetrachloride.

**Sicilian Oil.**—Petroleum was used, under this name, for illuminating purposes at Agrigentum, Sicily, before the beginning of the Christian era.

**Siegburgite.**—A fossil resin from the brown coal formation in the neighborhood of Troisdorf and Siegburg, near Bonn, Germany; it varies in color from golden yellow to brownish red, and is partially soluble in alcohol and ether (LASAULX, *Jahrb. Min.*, **1875**, 128; KLINGER and PITSCHKI, *Ber.*, **17** (1884), 2742).

**Signal Oil.**—A signal oil which has been recommended is composed of 20 per cent. of white fish oil and 80 per cent. of *mineral seal oil.*

**"Silicate of Carbon."**—A name for *lampblack.*

**Simetite.**—A resin near succinite from near Mt. Etna, Sicily; it is remarkable for its deep red color, contains but 0.4 per cent. of succinic acid, and has a specific gravity of from 1.052 to 1.068 (HELM and COMVENTZ, *Schriften Ges. Danzig*, **5** (1881), 293; **7** (1891), 198).

**Slab Oil.**—The following two neutral oils are said to be suitable:

| Gravity | Flash-point | Fire test | Cold test | Saybolt viscosity |
|---|---|---|---|---|
| 32¾°Bé. | 415°F. | 480°F. | 20°F. | 200 |
| 35° | 340° | 400° | 20° | 70 |

**"Slack Wax."**—A name for a mixture of paraffin wax and oil.

**Slate-Coal.**—A class of brown coal, dense, slaty, brownish black to black in color, and of uneven fracture.

See *Lignite.*

**Slide Oil.**—*Summer black oil* is recommended as a slide oil.

**"Slop Oil."**—Any liquid product of petroleum which is not up to quality. "Slop oils" are usually put aside for redistillation.

**Sludge.**—A term applied to the tar from the agitators in the chemical treatment of distillates. See pages 460, 584 and 591.

"Sludge acid" is sulphuric acid which has been used in the treatment of a petroleum distillate.

**Slushing Oil.**—*Summer black oil* is recommended as a slushing oil.

**"Smudge Oil."**—See *Orchard Heating Oil.*

**S. O. Co. Binder "B."**—This "blown" oil originates in petroleum from the Mid-Continent field; it contains 99.8 per cent. of bitumen soluble in carbon disulphide, 76.1 per cent. of bitumen soluble in 88°Bé. naphtha, 51.7 per cent. of pure bitumen as saturated hydrocarbons, 3.1 per cent. of paraffin scale, 0.68 per cent. of sulphur, and 12.6 per cent. of ash-free residual coke.

**S. O. Co. Binder No. 3.**—This "blown" oil originates in petroleum from the Mid-Continent field; it contains 98.8 per cent. of bitumen soluble in carbon disulphide, 74.9 per cent. of bitumen soluble in 88°Bé. naphtha, 44.3 per cent. of pure bitumen as saturated hydrocarbons, 1 per cent. of paraffin scale, 0.77 per cent. of sulphur, and 13.2 per cent. of ash-free residual coke.

**"Soft Paraffin."**—See *Petrolatum.*

**"Soft Petroleum Ointment."**—See *Petrolatum.*

**Soft Pitch.**—Pitch showing a penetration of more than 10.

**Solar Oil.**—1. A name given to *gas oil* from petroleum of the Gulf or Mid-Continent field; it is generally exported to Great Britain.

2. A Russian petroleum product possessing a specific gravity above 0.850, but not exceeding 0.880, and a flash-point not below 80°C. It is usually of a very pale yellowish color. Crude Baku oil yields 4 per cent. of solar oil. See *Pyronaphtha.*

3. Illuminating oil from shale oil, marketed in two grades: *prima*, possessing a specific gravity of 0.825 to 0.830 and boiling at 175° to 180°C.; and *secunda*, which has a density of 0.830 to 0.835 and boils at 195° to 200°C.

**"Solene."**—See *gasoline* and *petroleum ether*, with both of which the term is synonymous.

**Solid Bituminous Material.**—Bituminous material showing a penetration at normal temperature under a load of 100 grams applied for 5 sec. of not more than 10.

See *Bituminous Materials.*

**"Solidified" Gasoline.**—Rosculetz prepared "solidified" gasoline by dissolving stearic acid which had undergone prolonged treatment with hydrochloric acid at a high temperature, in the gasoline to be solidified and then mixing the solution with an alcoholic solution of sodium hydroxide at about 175°F. (*J. Ind. Eng. Chem.*, **3**, 528).

**"Solidified" Kerosene.**—On the conversion of kerosene into a solid jelly for convenience of transport and its reconversion into liquid form, see Ditmar, *Zap. Imp. Russ. Tekhn. Obsch.*, **16** (1882), otd. 1, 1.

**"Solidified" Petroleum.**—The following are two of the best known methods which have been resorted to in attempting to "solidify"

petroleum for use in grates: (1) absorption of the oil by a porous material, preferably itself combustible, as dry peat; and (2) the production of a jelly-like emulsion by the addition of soap or of a fatty oil and alkali to form a soap.

**"Solvent Naphtha."**—Both coal-tar naphtha and wood naphtha are marketed under this name.

**Specular Coal.**—See *Pitch Coal.*

**Spindle Oil.**—The lighter portion of the petroleum distillates used for lubrication, as Russian lubricating oil of the density 0.885 to 0.895, suitable for light-running machinery such as that used in cotton-spinning.

According to American practice, spindle oils range in gravity from 26° to 35°Bé., in flash from 320° to 450°F. (Cleveland open cup), in viscosity from 400 to 40 sec. Saybolt at 70°F., in cold test from 10° to 40°F., and in color from almost colorless to dark red.

Spindle oils are usually decolorized by filtration through fullers' earth or bone-black; they differ from paraffin oils in that for a given viscosity they are of higher Baumé gravity. For illustration, a spindle and a paraffin oil of the same gravity may have the following tests:

| | Spindle oil | Paraffin oil |
|---|---|---|
| Baumé gravity at 60°F..... | 30° | 30° |
| Flash, Cleveland open cup..... | 420°F. | 340°F. |
| Fire test, Cleveland open cup.. | 475°F. | 390°F. |
| Viscosity, Saybolt at 70°F.... | 300 sec. | 60 sec. |

Spindle oils are used for all kinds of high-grade lubrication, except steam cylinders: as high-grade engine oils; and for spindles in textile mills, whence the name, on account of the fact that spindle oil does not make bad stains on fabrics whereas paraffin oils do. They find wide application as automobile oils (*q.v.*), for which purpose they seem to be particularly well adapted. Specially purified oils of this class are being used in medicine with very successful results in treating cases of constipation [see *Petrolatum* (*Liquid*)].

Ordinary spindle oil is usually about the same grade as the non-viscous neutral oil described under *concrete form oil*, and stainless spindle oil is identical with 34° and 34.5° Bé. *sewing machine oil.*

Light spindle oil adapted for high-speed submerged cotton mill spindles should stand a 10°F. cold test.

**Splint Coal.**—The splint coal, or hard coal, of England ignites less readily than cherry coal (*q.v.*) and burns less rapidly.

**Squegee, or Squeegee.**—A tool with a rubber or leather edge for scraping or cleaning hard surfaces, or for spreading and distributing liquid material over and into the superficial interstices of roadways.

**Squegee Coat.**—An application by means of the squegee.

**"St. Quirinus Oil."**—Petroleum was used medicinally in Germany as

early as 1436 under the name "St. Quirinus Oil," the supply coming from the Tegernsee district of Bavaria.

**"Stainless Oils."**—*Spindle* or *loom oils* mixed with usually lard or neatsfoot oils. One well-known "stainless oil" is compounded of 40 per cent. neutral oil, 30 per cent. cottonseed oil, 20 per cent. olive oil, and 10 per cent. first-pressing castor oil.

**Standard White Oil.**—1. Standard white in color. The name is usually applied to *export oil.*

2. A Russian kerosene which has a specific gravity of 0.808 to 0.812 and is standard white in color.

See *Prime White Oil.*

**Stanekite.**—Stanekite is separated from *pyroretin* by boiling alcohol, which leaves it behind; it is insoluble in potassium hydroxide solution.

**"Stanolax."**—A proprietary name for liquid petrolatum, *q.v.*

**Steam Cylinder Oil.**—See *Cylinder Stocks.*

**Steuart Shale.**—See *Mungle Shale.*

**Still Coke.**—The residue left in the still on distilling crude shale oil to dryness. In the case of the best crude oil, it represents about 3 to 4 per cent. of the oil. It is used as a fuel, as carbon for electrical purposes, and for making moulders' blacking. Cf. *Coke.*

**Still Grease.**—The amorphous distillate from the end of the crude oil and heavy oil distillation in the shale-oil industry. It is used for grease-making.

**"Stink Coal."**—See *Dysodile.*

**"Stinkstone."**—See *Anthraconite.*

**Stitching Oil.**—A lubricant employed for stitching shoes; it is usually supplied in three grades: "water-white," for stitching white or light colored shoes; "fine white," and light strawcolored.

**"Stove Distillate."**—1. A stove gasoline before receiving a finishing treatment.

2. A product from California petroleum; it possesses a gravity of 30° to 34°Bé.

**Stove Gasoline.**—Gasoline (*q.v.*) used for gasoline stoves and making gas for illuminating purposes.

**Straight-Run Pitch.**—A pitch (*q.v.*) run in the initial process of distillation, to the consistency desired, with subsequent fluxing.

**"Straw Oil."**—A product possessing a gravity of 32.1°Bé., a Saybolt viscosity of 101 at 100°F., a flash-point of 365°F., and a cold test of 38°F. It has an initial boiling-point of 660°F. and a final boiling-point of 750°–800°F. It usually contains about 2.2 per cent. of paraffin wax.

**Suberane.**—For a discussion of the question, does suberane exist in petroleum, see MARKOVNIKOV, *J. Russ. Phys.-Chem. Soc.*, **34** (1902), 904 and 917.

**Succinellite.**—Succinic acid ($C_4H_6O_4$) obtained in orthorhombic crystals from *succinite*, of which it constitutes 2.5 to 6 per cent.

**Succinite.**—Succinite or amber occurs on the Prussian coast of the Baltic, in England, Denmark, and Russia; it is found in irregular masses, without cleavage, possesses a specific gravity of 1.050 to 1.096, and fuses at 250° to 300°C. On its properties, see HELM, *Schriften Ges. Danzig*, **7** (1891), 192; and DANA'S "Descriptive Mineralogy," 6th ed., 1002.

**Summer Black Oil.**—A black lubricating oil of 540°F. fire test, used as a heavy tempering oil and for waterproofing cement. See *Axle Oil.*

**"Summer Oil."**—A heavy railway car and engine oil which has a flash-point of above 140°C. and solidifies below −5°C.

**Sunned Oil.**—The trade-name of crude petroleum which was sometimes increased in density and fitted for use as a lubricant by exposing it to the sun as a thin layer on the surface of a tank of warm water, the more volatile portions being thus in part removed by evaporation. See page 527.

**Superficial Coat.**—A light surface coat in pavement construction.

**Surface Coat.**—See *"Carpet."*

**Surface Treatment.**—Treating the finished surface of a roadway with bituminous material.

**Surfacing.**—(1) The crust or pavement. (2) Constructing a crust or pavement. (3) Finally finishing the surface of a roadway. (4) Treating the surface of a finished roadway with a bituminous material.

**Tabbyite.**—See *Wurtzilite.*

**"Tailings."**—See *Residuum.*

**Tar.**—Bitumen which yields pitch upon fractional distillation and which is produced as a distillate by the destructive distillation of bitumens, pyrobitumens, or organic material. See *Pitch.*

Tar is also defined as a dark colored, bituminous substance, liquid or semi-liquid at room temperature, often possessing a characteristic "tarry" odor, usually insoluble in water, but miscible with carbon disulphide, benzol, etc., and which on distillation, oxidation, etc., forms a pitch. Its composition and origin are variable.

*Natural tars:*

1. From the distillation of petroleum (petroleum tar or soft petroleum asphalt).

2. From the destructive distillation of organic substances and pyrobitumens (pine tar, wood tar, bone tar, coal tar, mineral tar (maltha), petroleum tar, etc.).

**"Tar Distillate."**—The paraffin distillate, or distillate containing the paraffin wax. See page 462.

**Tasmanite.**—A reddish brown, resinous mineral, disseminated in scales through a laminated shale ("combustible shale") from the river Mersey, on the north side of Tasmania; it has the specific gravity 1.18 and yields oil on distillation.

On this mineral, see CHURCH, *Phil. Mag.*, (4), **28** (1864), 465; NEWTON,

*Geol. Mag.*, **2** (1875), 336; and STEPHENS, *Proc. Roy. Soc. Tasmania*, March, **1876**, 5.

**Tempering Oil.**—A viscous neutral oil, red in color, which, when straight reduced, has a gravity of 30°Bé., a flash-point of 415°F., a fire test of 480°F., a cold test of 20°F., and a Saybolt viscosity of 220; and, when stock-loaded, a gravity of 30.5°Bé., a flash-point of 395°F., a fire test of 450°F., a cold test of 30°F., and a Saybolt viscosity of 240.

A steam-refined cylinder stock equivalent to 26°Bé. *Hammer oil* is used as a heavy tempering oil, and *summer black oil* has been recommended for the same purpose.

**"Terralbolia."**—A proprietary name for liquid petrolatum, *q.v.*

**"Terraline."**—A proprietary name for liquid petrolatum, *q.v.*

**"Testefas."**—A Russian illuminating oil which possesses a specific gravity of 0.820 to 0.823 and a flash-point of not below 38°C.

**Tetracosane.**—Tetracosane ($C_{24}H_{50}$) has been separated from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565). MABERY (*ibid.*, **40** (1904), 349) also separated tetracosane from the solid hydrocarbons that collect in certain oil wells in Pennsylvania and from commercial paraffin.

**Tetradecane.**—Tetradecane ($C_{14}H_{30}$) has been isolated from Pennsylvania petroleum (MABERY, *Proc. Am. Acad.*, **37** (1902), 565).

**Tetratricontane.**—This hydrocarbon ($C_{34}H_{66}$) has been separated by MABERY (*Proc. Am. Acad.*, **40** (1904), 349) from the solid hydrocarbons that collect in certain oil wells in Pennsylvania.

**Thallene.**—See account given under *petrocene*.

**"Thickened Oils."**—Mineral oils thickened by dissolving in them small amounts of unvulcanized rubber or of aluminum soap; they are intended for certain lubricating purposes.

**Thiophene.**—Thiophene ($C_4H_4S$) was found by KHARITCHKOV (*J. Russ. Phys.-Chem. Soc.*, **31**, 655) in Grozni petroleum to the extent of 1 part in one million; the same investigator reported its presence in benzol to the extent of 1 part per ten million.

**Thread Cutting Oil.**—See *engine oil*, with which this viscous neutral oil is identical.

Pipe thread cutting oil is sometimes compounded of 60 to 80 per cent. of *engine oil* and 40 to 20 per cent. of lard oil, while *engine oil* alone is used for cutting screws, bolts and nuts.

**300° Burning Oil.**—An oil of 300°F. fire test and 265°F. flash-point, possessing a density of 0.825–0.830. Under the name of "mineral sperm oil," "mineral colza oil," or "mineral seal oil," it is used in cases wherein the employment of a more readily ignitable oil might be objectionable. About 10 per cent. of such an oil may be obtained from Pennsylvania petroleum.

**Tiffanyite.**—A name proposed by KUNZ (*Trans. N. Y. Acad. Sci.*, **14** (1895), 260) for a hydrocarbon assumed to be present in certain dia-

monds, namely, those which, on this account, exhibit fluorescence and phosphorescence.

**Toluene.**—Toluene ($C_7H_8$) has been found to be invariably present in petroleum. See pages 6 and 570.

**"Tops."**—The "No. 1 Tops" from California petroleum includes gasoline stock; the usual color is water-white and the gravity is about 55°Bé.

**Torbanehill Mineral.**—Torbanehill mineral was evidently the most important of the first bituminous minerals obtained from the Scotch fields. Its characters were as follows:

The Torbanehill mineral was of a brown or nearly black color; there were brown and black varieties, and the brown was the richer. It had a yellow or fawn-colored streak, without luster, and sub-conchoidal fracture, was amorphous, and apparently homogeneous when fresh, but showed distinct stratification when spent in the retort. It was very indestructible and did not deteriorate with weathering. Near basalt dikes it became soft, sticky and brown, resembling melted India rubber. In the laboratory, it took fire readily when put to a flame, split, did not fuse, burned with a very smoky flame, gave an empyreumatic odor, and left a considerable amount of white ash.

The crude oil taken from it was 96 to 130 gal. per ton, specific gravity about 0.890. It was richer in the east part of the field than the west, and it deteriorated somewhat in depth. It yielded 44 to 48 per cent. of crude oil, and 1 to 1½ per cent. of solid paraffin. Heated in a closed crucible, it gave 60 to 70 per cent. of volatile hydrocarbons; 6.6 to 13.3 per cent. of fixed carbon, and 12.8 to 23.2 per cent. of ash. The specific gravity varied from 1.17 to 1.316.

**Torbanite.**—See *Torbanehill Mineral.* On the production of mineral oil from the torbanite of New South Wales, see PETRIE *J. Soc. Chem. Ind.*, **24** (1905), 996.

**Torch Oil.**—The product described under *paint oil* is also used as a torch oil.

**Transformer Oil.**—The main requirements for oils for high-tension electrical transformers are freedom from water and mineral acids; further, they should show little or no volatility at 100°C. Those machine oils derived from petroleum which have a flash-point of over 160°C. (open test), with a volatility of less than 0.1 per cent. in 5 hr. at 100°C., provided they satisfy the electrical requirements, are suitable for use in transformers.

An extra pale lemon, non-viscous neutral oil which is largely used for transformers possesses a gravity of 34°Bé., a flash-point of 340°F., a fire test of 400°F., a cold test of 20°F., and a Saybolt viscosity of 80.

On transformer oils, see BRETH, *Petroleum*, **7** (1911), 290; and DUCKHAM, *Electrician*, **67** (1911), 212.

**Transmission Oil.**—A steam-refined cylinder stock possessing a

gravity of 25°Bé., a flash-point of 610°F., a fire test of 710°F., a cold test of 30°F., and a Saybolt viscosity of 245.

**Tricosane.**—Tricosane ($C_{23}H_{48}$) has been separated from Pennsylvania petroleum by MABERY (*Proc. Am. Acad.*, **37** (1902), 565). The same investigator (*ibid.*, **40** (1904), 355) also separated tricosane from commercial paraffin.

**Tridecane.**—Tridecane ($C_{13}H_{28}$) has been isolated from Pennsylvania petroleum (MABERY, *Proc. Am. Acad.*, **37** (1902), 565).

**Trinidad Asphalt.**—Asphalt from the Pitch Lake or from deposits lying between the lake and the seacoast, on the island of Trinidad. See *Lake Pitch* and *Land Pitch*.

Trinidad asphalt contains 68.12 per cent. of bitumen and 31.88 per cent. of fixed carbon, etc. Its chemical composition has been given as follows:

| | |
|---|---|
| Carbon | 80.32 per cent. |
| Hydrogen | 6.30 per cent. |
| Nitrogen | 0.50 per cent. |
| Oxygen | 1.40 per cent. |
| Sulphur | 11.48 per cent. |
| | 100.00 per cent. |

Trinidad Lake asphalt contains 56.5 per cent. of bitumen soluble in carbon disulphide, 63.1 per cent. of bitumen soluble in 88°Bé. naphtha, 24.4 per cent. of pure bitumen as saturated hydrocarbons, 6.2 per cent. of sulphur, and 10.8 per cent. of ash-free residual coke.

See *Asphalt* and *Bituminous Materials*.

**Trinidad Pitch.**—The deposit of solid or semi-solid bitumen constituting the Pitch Lake of Trinidad.

**Trinkerite.**—A red to brown mineral which resembles *tasmanite* in composition, found in brown coal in Istria and Styria; it has a specific gravity of 1.025, fuses at 168° to 180°C., and is soluble in hot benzol (TSCHERMAK, *J. prakt. Chem.*, **2** (1870), 258).

**Turbine Oil.**—A mineral oil adapted for the lubrication of the heaviest types of horizontal turbines should possess a high flash test and low viscosity and should retain its body and efficiency under warm temperatures. It should be filtered to a bright yellow color and should not absorb moisture.

An oil having a gravity of 30°Bé., 150 sec. viscosity at 70°F., Saybolt, and 420°F. flash is said to have given good results in steam turbines.

On turbine oil, see JOB, *Proc. Am. Soc. Test. Mat.*, **9** (1909), 614; and SCHWARZ and MARCUSSON, *Mitt. k. Materialprüf.*, **32** (1914), 496.

**"Tur-Min-Tine."**—A petroleum substitute for turpentine.

**Turpentine Substitutes.**—These products are usually intermediate between gasoline and illuminating oil (49° naphtha). They vary in gravity from 40° to 58°Bé., and are said to be more homogeneous than

burning oils. As they are designed for paint thinners and for admixture with turpentine, they should evaporate without leaving residues or stains. A representative sample found in the market gave the following tests:

| | |
|---|---|
| Baumé gravity at 60°F | 48.2 |
| Refractive index at 60°F | 1.4595 |
| One drop evaporated from white paper | No stain. |
| 15 drops in a watch glass evaporated in | 2 hr., 23 min. |
| 15 drops of turpentine in watch glass evaporated in | 2 hr., 15 min. |
| Initial boiling point | 282°F. |
| Final boiling point | 426°F. |

A turpentine substitute termed "Tur-Min-Tine" has a gravity of 52°Bé. and a flash-point of 105°F.; 95 per cent. is said to distil between 147° and 220°C. See *"White Spirit."*

A Rotterdam, Netherlands, refinery has put on the market a substitute for turpentine that consists of certain aromatic hydrocarbons occurring in unusual abundance in the crude petroleum of Borneo and easily separated by fractionation.

**Turrelite.**—A Texas asphalt-rock, a shelly mass cemented together by bitumen. On its composition, see PECKHAM, *J. Soc. Chem. Ind.*, **16** (1897), 996.

**Uintaite.**—See *Gilsonite.*

**Unfiltered Stocks.**—The dark green unfiltered stocks from Pennsylvania petroleum have the following properties:

| Gravity | Flash-point | Fire test | Saybolt viscosity |
|---|---|---|---|
| 26°Bé. | 540°F. | 600°F. | 140 |
| 25½° | 585° | 650° | 200 |
| 25° | 600° | 660° | 230 |
| 24° | 630° | 700° | 270 |

See *Cylinder Stocks.*

**Unguentum Paraffinum.**—See *Petrolatum.*

**Unguentum Petrolei.**—See *Cosmoline.*

**Urpethite.**—A yellowish brown to brown mineral having the consistence of soft tallow and a specific gravity of 0.885; it melts at 39°C. and is readily soluble in cold ether. Urpethite constitutes about four-fifths of Urpeth Colliery ozokerite (JOHNSTON, *Phil. Mag.*, (2), **12** (1838), 389).

**"Usoline."**—A proprietary name for liquid petrolatum, *q.v.*

**Valve Oil.**—See *Cylinder Stocks.*

**"Valvoline."**—See *Viscosine.*

**Varnish Makers' Naphtha.**—Both deodorized naphtha (58° to 60°Bé.) and turpentine substitute of 52°Bé. gravity are used by varnish manufacturers.

**"Vaselene" or "Vaseline."**—See *petrolatum*, of which vaseline, or petroleum jelly, is a proprietary brand; but see especially page 631.

**Vaseline Oil.**—(1) A paraffin oil obtained from shale oil, used in Germany as a solvent for ceresin.

(2) Vaseline is obtainable not only from American oils (see page 632), but also from the oils of Galicia, Elsass (*Chem.-Zentr.*, **1882**, 319), and Russia (MENDELÉEFF, *Fortschr. Jahresb. Chem.*, **1882**, 1458; and BIEL, *ibid.*, **1882**, 1466). The Galician product has been exhaustively examined by ENGLER and BÖHM (*Dingler's polyt. J.*, **262** (1886), 468 and 524), who operated upon two particularly suitable crude oils sent them from Ropa. See page 636.

**"Vaselon."**—A mixture of margaron and stearon dissolved in mineral oil; it resembles petrolatum (*q.v.*) and is used as an ointment base.

**"Vasogen."**—"Oxygenated vaseline." A vehicle for applying iodine, ichthyol, salicylic acid, etc., in the treatment of skin diseases.

**Vegetable Oil Adulterant.**—See *Paint Oil.*

**"Viridine."**—See account under the caption *petrocene.*

**"Viscosine."**—A Russian lubricating oil which possesses a specific gravity of 0.925 to 0.935, a flash-point of 290° to 310°C., and an Engler viscosity of 5 min. at 100°C.

**Viscous Neutral Oils.**—See *Neutral Oil.*

**Volatile.**—Applied to those fractions of bituminous materials which will evaporate at climatic temperatures.

**Vulcan Oil.**—A product from petroleum having a specific gravity of 0.910 to 0.960 at 15°C.

On the tar-content of this oil, see LÖWY, *Chem. Rev. Fett- Harz-Ind.*, **17** (1910), 6; and on American vulcan oil, see OTT, *Dingler's polyt. J.*, **187** (1868), 171.

**Walchowite.**—A yellow, resinous oxygenated hydrocarbon which occurs in brown coal at Walchow, in Moravia; it has a specific gravity of 1.0 to 1.069, fuses to a yellow oil at 250°C., and forms a dark brown solution in sulphuric acid (MELION, *Lotos*, **5** (1855), 122).

**Waste Acid and Alkali.**—See pages 584 to 593.

**Watch Oil.**—The oil obtained from the dolphin, blackfish or "snuffer" is used for lubricating the delicate machinery in watches and clocks.

**Water-Bound.**—Bonded with the aid of water. See *Bond.*

**Water-Gas Tars.**—Tars (*q.v.*) produced by cracking oil vapors in the manufacture of carbureted water-gas.

**Water-Soluble Oils.**—These have the property of forming permanent emulsions or almost clear solutions with water. They are prepared by dissolving ammonia-, potash-, or soda-soaps of oleic, sulpho-fatty, rosin, or naphthenic acids in mineral oils; but sometimes ammoniacal

liquor, naphtha or alcohol is added, and in certain cases the rosin oils are first air-blown. The resulting water-soluble products, which may be regarded as colloidal solutions of mineral oil in an acid soap solution, are employed as lubricants in boring, lathe-cutting, milling, and polishing operations, and for dressing wool-fiber in spinning and weaving and for laying dust.

See *Westrumite.*

**Water-White Oil.**—A high-grade illuminating oil, water-white in color, usually obtainable in three grades, viz.:

| Gravity | Fire test |
|---|---|
| 45°Bé. | 170°F. |
| 47° | 150° |
| 48° | 120° |

See *Illuminating Oils* and *Kerosene.*

**"Water-White Mineral Oil."**—A name for liquid petrolatum, *q.v.*

**Wax.**—A term applied to unctuous, fusible, and more or less viscous to solid substances, having a characteristic "waxy" luster, and which are insoluble in water, but more or less soluble in carbon disulphide, benzol, etc. They are extremely susceptible to changes in temperature. Their composition, origin and color are variable.

(*a*) Natural—
1. Mineral (*e.g.*, ozokerite, ceresin, montan wax, hatchettite, etc.).
2. Vegetable (*e.g.*, Japan wax, carnauba wax, etc.).
3. Animal (*e.g.*, beeswax, spermaceti, etc.).

(*b*) Artificial—
1. From the distillation of paraffin-base petroleum, ozokerite, etc.
2. From the destructive distillation of lignite (brown coal), paraffinaceous shales, etc.

See *Paraffin Wax.*

**Wax Coal.**—See *Earthy Brown Coal.*

**"Wax Distillate."**—Neutral oil distillate before the separation of paraffin wax. See *Neutral Oil.*

**Wax Tailings.**—This product of decomposition contains chrysene, picene and other compounds formed by destructive distillation. It is of dark green color but darkens on exposure to light. It is asphaltic in nature and varies in melting point according to the care with which it is separated and later refined. It is used for weather- and waterproofing compounds, in some cases as a flux in street-paving mixtures, and as a filler in very cheap axle greases. See page 461.

**Westrumite.**—In 1903, Westrum patented the use of a "soluble oil," such as that of Boleg, in admixture with a large quantity of water, for the sprinkling of roads with the object of "laying" dust. Many

stretches of road have been treated with "westrumite," as the product is termed, especially in connection with motor-car races, and the "laying" of the dust has been found to be thus effected with greater permanence than by the employment of water, the presence of the oil apparently arresting evaporation.

**"Wet" Natural Gas.**—(1) Natural gas from which gasoline can be extracted in quantity sufficient to warrant the installation of a plant.

(2) Natural gas which contains readily condensible gasoline. Cf. *"Dry" Natural Gas.*

**Wheelerite.**—A yellowish resin, found in the Cretaceous beds of northern New Mexico, filling the fissures of the lignite, or interstratified in thin layers in it; it is soluble in ether. Described by LOEW in *Am. J. Sci.*, (3), **7** (1874), 571.

**"White Liquid Vaseline."**—A name for liquid petrolatum, *q.v.*

**"White Naphthene Oil."**—A name proposed for pharmaceutic oil, or *liquid petrolatum.*

**"White Oil."**—An odorless and colorless oil possessing a specific gravity of 0.857. It is used medicinally and as a base for creams, salves and ointments. See *Oil of Paraffin* and *Petrolatum (Liquid)*.

**"White Paraffin Oil."**—A name for liquid petrolatum, *q.v.*

**"Whiteruss."**—A name for liquid petrolatum, *q.v.*

**"White Spirit."**—The turpentine substitutes used in the manufacture of varnishes, sold in England as "white spirit," have generally a flash-point above 23°C., mostly at 26.7°C., yield 90 per cent. of distillate up to 140°C., have a specific gravity of about 0.785, and are characterized by a mild odor.

See *Turpentine Substitutes.*

**"White Vaseline."**—A proprietary brand of *Petrolatum (White)*.

**"Winter Oil."**—A heavy railway car and engine oil which has a solidifying point of below −20°F.

See *Car Oil.*

**Wollongongite.**—A name given provisionally to a supposed hydrocarbon from Wollongong, New South Wales. It has been shown to be simply *kerosene shale* (*q.v.*). See SILLIMAN, *Am. J. Sci.*, (2), **48**, 85.

**Wool Stocks.**—Three wool stocks are made from Pennsylvania petroleum:

| Gravity | Flash-point | Fire test | Saybolt viscosity |
|---|---|---|---|
| 31.5°Bé. | 400°F. | 450°F. | 155 |
| 32.4° | 390° | 440° | 140 |
| 32.5° | 375° | 425° | 140 |

Sun-bleached neutral oil may be compounded with lard oil to make a rich, creamy emulsion adapted as a wool oil.

**Worm Gear Oil.**—The product described under *transmission oil* is said to be satisfactory for worm gears.

**Wurtzilite.**—A Utah asphaltic mineral which is jet-black by reflected light and deep red in thin plates; it softens in hot water, toughens, and becomes more elastic; in a candle flame it softens and burns with a bright flame; it is practically insoluble in gasoline of 76°Bé., partly soluble in ether, carbon disulphide and turpentine, and less so in carbon tetrachloride. See BLAKE, *Eng. Min. J.*, **48**, (1889), 542; **49**, 106; and TAYLOR, *Salt Lake Min. Rev.*, **18**, No. 19, 43.

**Xylenes in Petroleum.**—Xylene ($C_8H_{10}$) was found in Rangoon oil by DE LA RUE and MÜLLER, and in Pennsylvania oil by SCHORLEMMER; isoxylene in Galician oil by PAWLEWSKI and LACHOWICZ, in Caucasian oil by KRAMER, and in that of Baku especially by MARKOVNIKOV. Paraxylene (boiling point, 137°C.) has been isolated from Galician oil by PAWLEWSKI (*Ber.*, **18** (1885), 1915).

**Xyloretinite.**—A white resin fusing at 165°C., derived by FORCHHAMMER (*J. prakt. Chem.*, **20** (1840), 459) through the action of alcohol on fossil pine-wood from the marshes of Holtegaard in Denmark. See *Hartine.*

**"Yellow Ozokerine."**—A product resembling vaseline, but less homogeneous, produced from crude *ozokerite;* it is used in ointments and pomades.

**Yellow Wax.**—A viscous, semi-solid, difficultly volatile substance obtained on distillation of still residuum. It contains anthracene ($C_{14}H_{10}$) and other hydrocarbons of complex structure.

See *Residuum.*

**Zietrisikite.**—A mineral resembling ozokerite in most physical characters and in composition, but almost completely insoluble in ether and melting at 90° C.; it occurs in Moldavia (MAGNUS, *Ann. chim. phys.*, (2), **55**, 217; MALAGUTI, *idem*, (2), **63**, 390; *Compt. rend.*, **4** (1837), 410).

# INDEX[1]

"An index is the bag and baggage of a book."

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